federal register

TUESDAY, JUNE 8, 1976

PART II:

ENVIRONMENTAL PROTECTION AGENCY

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STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES

Amendments to Reference Methods

ENVIRONMENTAL PROTECTION AGENCY

[40 CFR Part 60]

[FRL 536-4]

STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES

Proposed Amendments to Reference Methods

On December 23, 1971, the Environmental Protection Agency promulgated standards of performance for five categories of stationary sources under section 111 of the Clean Air Act, as amended. An appendix to the regulation contained Reference Methods 1–9, which detailed requirements for performance testing of stationary sources. Since promulgation of these reference methods EPA has continued to evaluate them. As a result, the need for a number of changes which would clarify the methods and/or improve their accuracy and reliability has become apparent. The following proposed amendments incorporate these changes to Reference Methods 1–8. Revisions to Reference Method 9 were promulgated on November 12, 1974 (39 FR 39872).

Changes common to all eight of the reference methods are: (1) the clarification of procedures and equipment specifications, and (2) the addition of metric units along with English units. Specific changes to the methods are:

METHOD 1

A statement was added to clarify that the method does not apply to stacks containing cyclonic or swirling flow or stacks smaller than 0.3 m (1 ft) in diameter or 0.07 m^2 (0.8 ft^2) in cross sectional area. A procedure for verifying the existence of non-cyclonic or non-swirling flow was added. For cases where large cross sectional variation of the pollutant concentration is suspected or for unusually large diameter stacks, the method was revised to provide that more than two traverse diameters may be specified by the Administrator.

METHOD 2

The use of the method has been limited to non-cyclonic or non-swirling gas streams. Greater details for calibration of the Type S pitot tube have been added including: criteria for standard type pitot tubes; specification of calibration at 915 m/min (3000 ft/min); details of acceptable wind tunnel systems; and additional details for calibrating isolated pitot tubes and pitobe assemblies.

METHOD 3

For determining the molecular weight of a stack gas sample, it is now acceptable to use either an Orsat analyzer or a Fyrite [1] type combustion analyzer. Previously, only the Orsat analyzer was specified. The integrated gas-sampling train for this method was altered to include a surge tank before the rate meter in order to eliminate pulsation effects caused by the diaphragm pump. Also, because this method requires proportional sampling, an inclined manometer was added to the train to measure velocity head.

Where low CO_2 (less than 4%) or high O_2 (greater than 15%) concentrations exist, the procedure has been revised to require an Orsat having at least 0.1% subdivisions. The revised method also provides sampling site selection criteria and criteria for determining the number of sample points. More detail has been added to the analytical procedure. Finally, the former criteria for three consecutive measurements have been changed to require three measurements within 0.3% for greater than 3% CO_2 and 0.2% for less than 3% CO_2.

METHOD 4

This method now contains two separate methods for moisture determination: (1) a reference method for cases where the Method 5 train is not used, and (2) an approximation method for moisture content to be used for setting isokinetic sampling rates. In the moisture sampling train by the approximation method, the rate meter is now located before the dry gas meter.

METHOD 5

The specification for temperatures around the filter holder was revised to read "no greater than 120±14° C (248±25° F), or such other temperature as specified by an applicable subpart of the standards." The revised wording of the temperature specification does not change the procedure contained in the original method; it only clarifies the intended procedure by providing more specific instruction. The revised language also provides flexibility for the Administrator to specify other temperature limits in applicable subparts of the standards. Method 5 employs an out-of stack filter to facilitate temperature control. This usage is not changed by these proposed amendments. Specifications for weight and volume measurements were changed to reflect the capabilities of most widely used apparatus. To further insure the validity of the sample, leak checks of the sampling train are now required after sampling runs as well as before. Finally, the gas-sampling train was altered to include a stack gas temperature sensor.

METHOD 6

In the sampling train, the flow control valve is now located before the pump instead of after to allow better leak checks. Samples collected by the train are to be diluted to 100 ml instead of 50 ml to allow the number of rinses of the impingers necessary for adequate sample recovery. The average flow rate through the sampling train was reduced to 1 liter/min to prevent reagent carry-over from one impinger to the next.

METHOD 7

A provision was added to require the potassium nitrate used for preparation of the standard solution to be dried at 105–110° C for a minimum of two hours. Currently, during sample recovery, sodium hydroxide is added to the sample solution. These revisions require that only enough sodium hydroxide be added to adjust the pH to 9–12. This will prevent a large excess of sodium hydroxide. Similarly, during the analysis procedure, only enough ammonium hydroxide may be added to the sample to raise the pH to 10. This requirement prevents possible differences in color intensity due to an excess of ammonium hydroxide. Also during sample analysis, only one-half of the sample is to be analyzed to avoid loss of the sample due to analytical error. Finally, two changes concerning the spectrophotometer were made: (1) for spectrophotometer calibration, an equation is provided to determine a factor that insures the best fit through the calibration points, and (2) the absorbance measurement is now to be made at 410 nm instead of 420 nm.

METHOD 8

During sample analysis a 10 ml aliquot of SO_2 sample is specified instead of 25 ml to reduce the amount of titrant required. A stack gas temperature sensor was added to the integrated gas-sampling train.

Finally, EPA is presently in the process of converting the units in its standards to the International System of Units (SI). In keeping with this policy, we will soon convert the equipment specifications and procedures of the reference methods to SI. We anticipate that in some situations it will be necessary, for practical application, to use a mixture of SI and metric units. We solicit any comments that will expedite and facilitate this transition.

[1] Mention of trade names is not intended to constitute endorsement by EPA.

By this notice, the Administrator is inviting comments on the proposed revisions. Submittals should, wherever possible, be supported with data and/calculations.

Comments on the proposed revisions should be submitted, in triplicate, to the Emission Standards and Engineering Division, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, Attention: Mr. Don R. Goodwin. All comments post-marked no later than July 23, 1976 will be considered.

Copies of comments received will be available for public inspection during normal business hours at the Public Information Reference Unit (EPA Library), Room 2922, 401 M Street, SW., Washington, D.C.

This amendment is proposed under the authority of section 111 of the Clean Air Act, as amended (42 U.S.C. 1857c6).

Dated: May 27, 1976.

JOHN QUARLES,
Acting Administrator.

It is proposed to amend Part 60 of Chapter I of Title 40 of the Code of Federal Regulations by revising Methods 1 through 8 of Appendix A—Reference Methods as follows:

APPENDIX A—REFERENCE METHODS

METHOD 1—SAMPLE AND VELOCITY TRAVERSES FOR STATIONARY SOURCES

1. *Principle and Applicability.*

1.1 Principle. A sampling site and the number of traverse points are selected to aid in the extraction of a representative sample.

1.2 Applicability. This method is applicable to sampling of gas streams contained in ducts, stacks, or flues.

It is intended that all new sources consider the requirements of this method before construction of the affected facility. Should they be overlooked, some sites may not lend themselves to this method and temporary alterations to the stack or deviation from the standard procedure may be required. Such cases are subject to approval by the Administrator.

This method is not applicable to stacks containing cyclonic or swirling flow (see § 2.4) or stacks smaller than about 0.3 m (1 ft) in diameter or 0.07 m^2 (0.8 ft^2) in cross sectional area. When these cases are encountered, an alternate procedure, subject to approval of the Administrator, is required.

2. *Procedure.*

2.1 Sampling site. Select a sampling site that is at least 8 stack or duct diameters downstream and 2 diameters upstream from any flow disturbance such as a bend, expansion, contraction, or visible flame. If impractical, select an alternate site that is at least 2 stack or duct diameters downstream and 0.5 diameter upstream from the flow disturbances. For a rectangular cross section, use an equivalent diameter calculated from the following equation to determine the respective distances:

$$D_e = \frac{2LW}{L+W} \quad \text{Equation 1-1}$$

where:

D_e = equivalent diameter

L = Length

W = Width

2.2 Minimum number of traverse points. When the 8 and 2 diameter criterion can be met, the minimum number of traverse points shall be 12 for stack diameters greater than 0.6m (24 in.) and 8 for stack diameters equal to or less than 0.6 m (24 in.).

When the 8 and 2 diameter criterion cannot be met, use Figure 1-1 to determine the minimum number of traverse points. To use this figure, first determine the distances from the chosen sampling location to the nearest upstream and downstream disturbances. Divide each distance by the diameter or equivalent diameter to determine the distance in terms of the number of duct diameters. Then, determine from Figure 1-1 the minimum number of traverse points that corresponds (1) to the number of duct diameters upstream and (2) to the number of diameters downstream. Select the higher of the two minimum numbers of traverse points, or a greater value, such that for circular stacks the number is a multiple of four, and for rectangular stacks, the number follows the criteria in section 2.3.2.

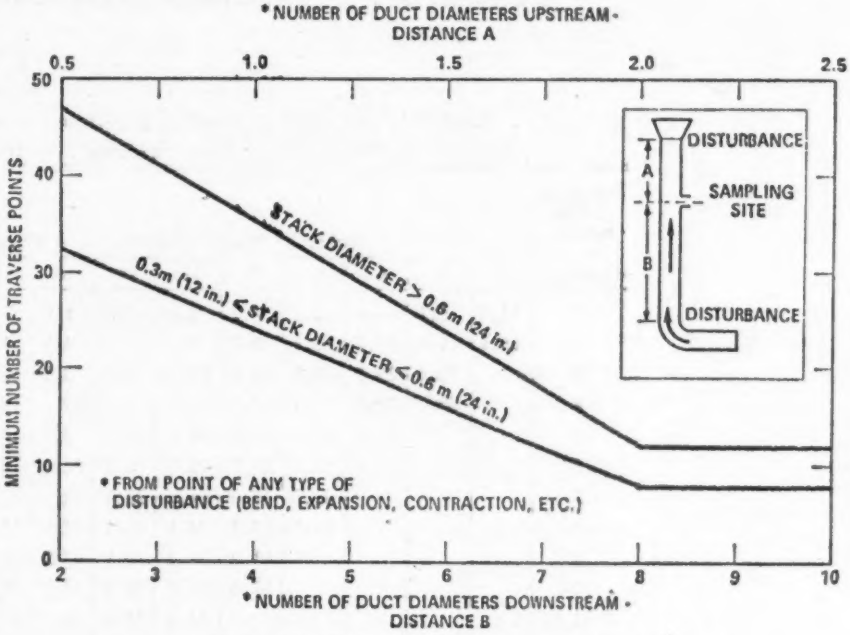


Figure 1-1. Minimum number of traverse points.

2.3 Cross sectional layout and location of traverse points.

2.3.1 Circular stacks. Locate the traverse points on two perpendicular diameters according to Table 1-1 and the example shown in Figure 1-2.

When large cross sectional variation of the pollutant concentration is suspected, the Administrator may specify that more than two diameters which divide the stack cross section into equal parts shall be used. More than two diameters may also be used with approval from the Administrator for unusually large diameter stacks.

One of the diameters shall be in a plane containing the greatest expected concentration variation, e.g., after bends one diameter shall be in the plane of the bend. This latter requirement becomes less critical as the distance from the disturbance increases. Therefore, other diameter locations may be used, subject to approval from the Administrator.

In addition, for stacks greater than 0.6 m (24 in.) no sampling points shall be selected within 2.54 cm (1 in.) of the stack walls, and for stacks equal to or less than 0.6 m (24 in.), no sampling points within 1.27 cm (½ in.) of the stack walls. To meet this criterion, do the following:

2.3.1.1 Stacks greater than 0.6 m (24 in.). When any of the traverse points, as located in section 2.3.1, fall within 2.54 cm (1 in.) of the stack walls, relocate them away from the stack walls to a distance of (1) 2.54 cm (1 in.) or (2) a distance equal to the nozzle inside diameter, whichever is larger. These relocated traverse points (on each end of a diameter) shall be the "adjusted" traverse points.

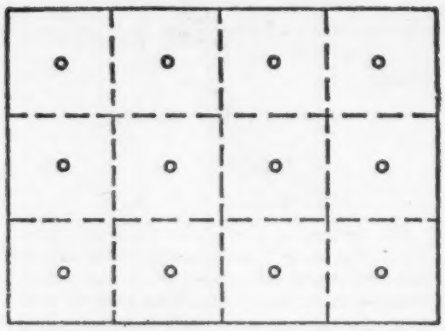


Figure 1-3. Example showing rectangular stack cross section divided into 12 equal areas, with traverse points at centroid of each area.

Table 1-1. Location of traverse points in circular stacks
(Percent of stack diameter from inside wall to traverse point)

Traverse point number on a diameter	Number of traverse points on a diameter											
	2	4	6	8	10	12	14	16	18	20	22	24
1	14.6	6.7	4.4	3.3	2.5	2.1	1.8	1.6	1.4	1.3	1.1	1.1
2	85.4	25.0	14.7	10.5	8.2	6.7	5.7	4.9	4.4	3.9	3.5	3.2
3		75.0	29.5	19.4	14.6	11.8	9.9	8.5	7.5	6.7	6.0	5.5
4		93.3	70.5	32.3	22.6	17.7	14.6	12.5	10.9	9.7	8.7	7.9
5			85.3	67.7	34.2	25.0	20.1	16.9	14.6	12.9	11.6	10.5
6			95.6	80.6	65.8	35.5	26.9	22.0	18.8	16.5	14.6	13.2
7				89.5	77.4	64.5	36.6	28.3	23.6	20.4	18.0	16.1
8				96.7	85.4	75.0	63.4	37.5	29.6	25.0	21.8	19.4
9					91.8	82.3	73.1	62.5	38.2	30.6	26.1	23.0
10					97.5	88.2	79.9	71.7	61.8	39.8	31.5	27.2
11						93.3	85.4	78.0	70.4	61.2	39.3	32.3
12						97.9	90.1	83.1	76.4	69.4	60.7	39.8
13							94.3	87.5	81.2	75.0	68.5	60.2
14							98.2	91.5	85.4	79.6	73.9	67.7
15								95.1	89.1	83.5	78.2	72.8
16								98.4	92.5	87.1	82.0	77.0
17									95.6	90.3	85.4	80.6
18									98.6	93.3	88.4	83.9
19										96.1	91.3	86.8
20										98.7	94.0	89.5
21											96.5	92.1
22											98.9	94.5
23												96.8
24												98.9

TRAVERSE POINT	DISTANCE, % of diameter
1	4.4
2	14.7
3	29.5
4	70.5
5	85.3
6	95.6

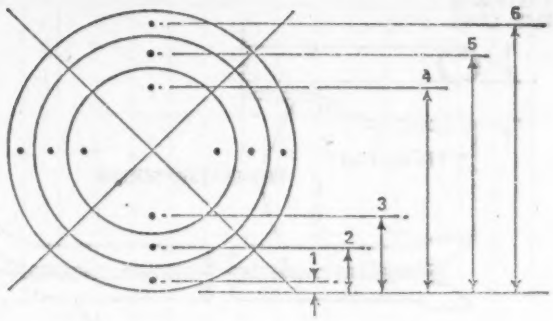


Figure 1-2. Example showing circular stack cross section divided into 12 equal areas, with location of traverse points at centroid of each area.

In some cases, two successive traverse points may need to be relocated to a single adjusted traverse point at each end of the traverse diameters. The sampling time at each of these "adjusted" traverse points shall be twice as long as at a "non-adjusted" sampling point as determined by the respective pollutant sampling method, e.g., Method 5.

2.3.1.2 Stacks equal to or less than 0.6 m (24 in.). Follow the procedure in section 2.3.1.1, noting only that the "adjusted" points should be relocated at 1.27 cm (0.5 in.) from the wall instead of 2.54 cm (1 in.).

2.3.2 Rectangular stacks. Divide the cross section into as many equal rectangular areas as traverse points as determined in sections 2.1 and 2.2 of this method, such that the ratio of the length to the width of the elemental areas is between one and two. Locate the traverse points at the centroid of each equal area according to the example in Figure 1-3.

The situation of section 2.3.1 concerning sampling points too close to the stack walls is not expected to arise with rectangular stacks. On the remote possibility that it does occur, consult with the Administrator.

2.4 Verification of non-cyclonic or non-swirling flow. Generally, cyclonic or swirling flow can be expected after such devices as cyclones and inertial demisters that follow venturi scrubbers or in stacks that have tangential inlets or two inlets that are opposite each other. At times, cyclonic flow can be detected by visual observations of the effluent plume. However, a Type S pitot tube as described in Method 2 can be used as a tool to verify the presence of cyclonic or swirling flow by doing the following:

2.4.1 Level and zero the manometer. Position the Type S pitot tube at each of the traverse points such that the face openings are perpendicular to the stack cross-sectional plane. A null (zero) reading at "0° reference" denotes the absence of cyclonic flow. If the reading is not zero, then rotate the pitot tube about ±10°. A null reading within the limits of ±10° rotation from 0° reference indicates an acceptable flow condition.

2.4.2 Conduct a velocity traverse according to Method 2. If there are negative velocity pressure readings, unacceptable flow conditions exist.

2.4.3 When unacceptable flow conditions are encountered, alternate procedures, subject to approval of the Administrator, are required.

3. *References.*

3.1 Determining Dust Concentration in a Gas Stream, ASME Peerformance Test Code #27, New York, N.Y., 1957.

3.2 Devorkin, Howard, et al., Air Pollution Source Testing Manual, Air Pollution Control District, Los Angeles, Calif., November 1963.

3.3 Methods for Determination of Velocity. Volume, Dust and Mist Content of Gases, Western Precipitation Division of Joy Manufacturing Co., Los Angeles, Calif. Bulletin WP-50, 1963.

3.4 Standard Method for Sampling Stacks for Particulate Matter, In: 1971 Book of ASTM Standards, Part 23, Philadelphia, Pa. 1971. ASTM Designation D-2928-71.

METHOD 2—DETERMINATION OF STACK GAS VELOCITY AND VOLUMETRIC FLOW RATE (TYPE S PITOT TUBE)

1. *Principle and Applicability.*

1.1 Principle. Stack gas velocity is determined from the gas density and from measurement of the velocity head using a Type S (Stausscheibe or reverse type) pitot tube.

1.2 Applicability. This method is applicable for measurement of the average velocity of a gas stream and for quantifying gas flow.

This procedure is not applicable for direct measurement in cyclonic or swirling gas streams. (Method 1, section 2.4 shows how to determine unacceptable flow conditions.) When these conditions exist, procedures such as the use of flow straightening devices must be employed subject to approval by the Administrator, to make accurate flow rate determinations.

2. *Apparatus.*

Specifications for the apparatus are given below. Any apparatus which has been demonstrated subject to approval of the Administrator, to be capable of meeting the specifications will be considered acceptable for the purposes of this method.

2.1 Pitot tube. Type S (Figure 2-1), or equivalent, calibrated according to the procedure in section 4. Other devices may be used when approved by the Administrator.

2.2 Differential pressure guage. Inclined manometer, or equivalent device, capable of measuring velocity head to within 10% of the minimum measured value or ±0.013 mm (0.005 in.), whichever is greater. Below a differential pressure of 1.3 mm (0.05 in.) water gauge, micromanometers with sensitivities of 0.013 mm (0.0005 in.) should be used. However, micromanometers may not easily be adaptable to the existing field conditions and are not easy to use with pulsating flow. Thus, alternative methods or other devices acceptable to the Administrator may be used when conditions warrant.

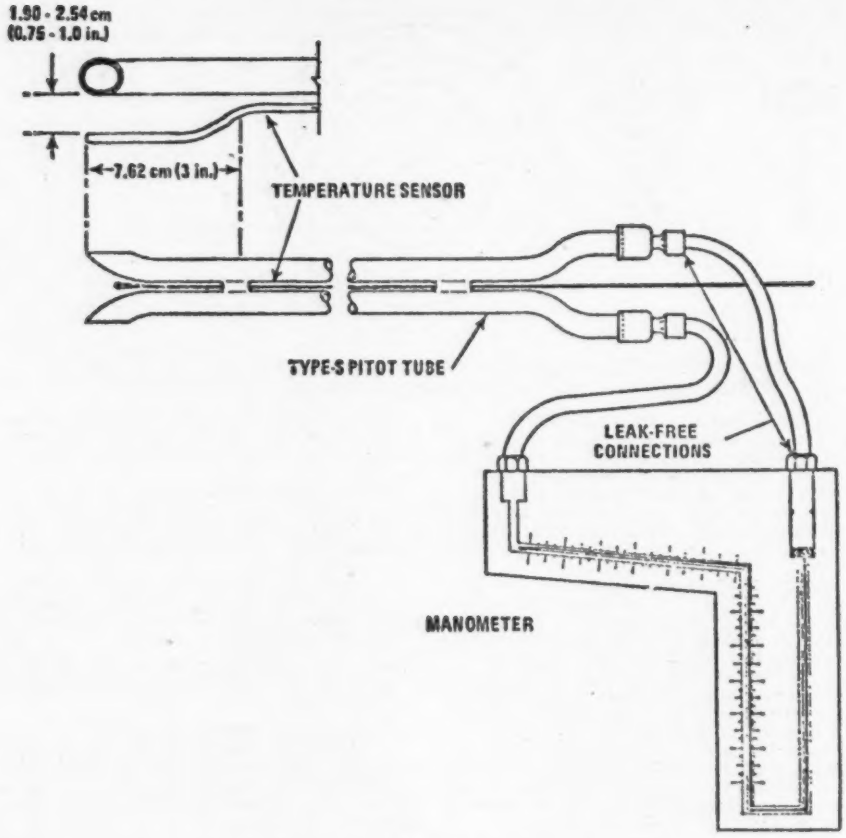


Figure 2-1. Pitot tube-manometer assembly.

The calibration of magnehelics, if used, must be checked on-site before and after each test run.

2.3 Temperature gauge. Thermocouple, liquid filled bulb thermometer, bimetallic thermometer, mercury-in-glass thermometer, or other gauges that are capable of measuring temperature to within 1.5% of the minimum absolute stack temperature. The temperature gauge shall be attached to the pitot tube such that the sensor does not touch any metal and its position is adjacent and about 1.90 to 2.54 cm (0.75 to 1 in.) from the pitot tube openings (see Figure 2–1). Alternate positions may be used if the pitot tube-temperature gauge system is calibrated according to the procedure of section 4. If it can be shown to the satisfaction of the Administrator that a difference of not more than 1% in the velocity measurement will be introduced, the temperature gauge need not be attached to the pitot tube.

2.4 Pressure probe and gauge. Piezometer tube and mercury- or water-filled U-tube manometer capable of measuring stack pressure to within 2.5 mm Hg (0.1 in. Hg). The static tap of a standard type pitot tube or one leg of a Type S pitot tube with the face openings positioned parallel to the gas flow may also be used as the pressure probe.

2.5 Barometer. Mercury, aneroid, or other barometers capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby weather bureau station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and the sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.6 Gas analyzer. To analyze gas composition for determining molecular weight. Use Method 3 or other methods specified by the Administrator for dry molecular weight and use Method 5 or Reference Method 4 for moisture content. Other methods may be used when approved by the Administrator.

2.7 Calibration pitot tube. Standard type, to calibrate the Type S pitot tube. The standard type pitot tube shall have a known coefficient obtained from the National Bureau of Standards, Route 70 S, Quince Orchard Road, Gaithersburg, Maryland. An alternative is to use a Prandtl type pitot tube designed according to the criteria (given below and illustrated in Figure 2–2; see also Reference 6.7 or 6.8 for greater detail) which ensure that its coefficient will be 0.99±0.01.

2.7.1 Hemispherical or ellipsodial tip (inlet end of the impact tube).

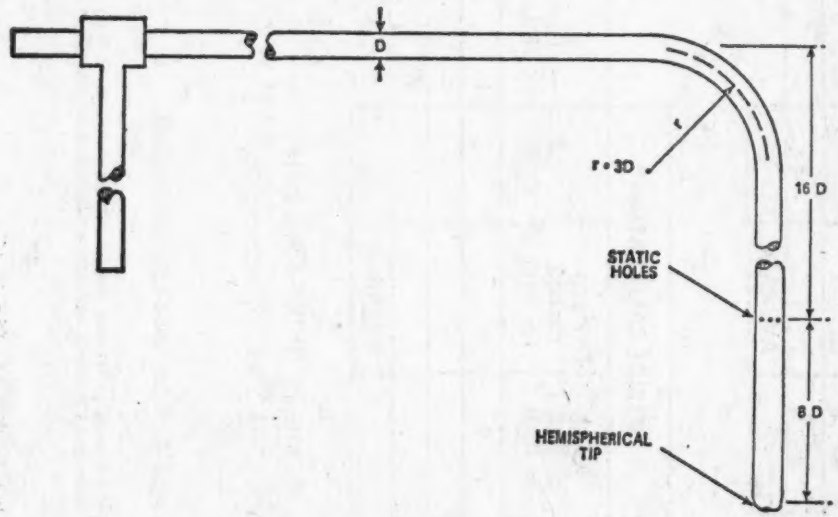


Figure 2-2. Standard Pitot tube.

2.7.2 Eight diameters of straight run (based on the diameter of the external tube) between the tip and the static pressure holes.

2.7.3 Sixteen diameters between the static pressure holes and the centerline of the external tube, following the 90° bend.

2.7.4 Eight static pressure holes of equal size (approximately 0.71 mm or 1/32 in. diameter), equally spaced in a piezometer ring configuration.

2.7.5 Ninety-degree bend of relatively large radius (approximately three diameters).

2.8 Calibration differential pressure gauge—For calibration purposes, inclined manometer, or equivalent device, capable of measuring velocity head to within 0.13 mm H_2O (0.005 in. H_2O).

3. *Procedure.*

3.1 Set up the apparatus as shown in Figure 2-1. Make sure all connections are tight and leak free. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the sample run. Record all necessary data as shown in the example data sheet (Figure 2-3).

3.2 Measure the velocity head and temperature at the traverse points specified by Method 1.

3.3 Measure the static pressure in the stack. One reading is usually adequate for all measuring points during the test; however, this must be confirmed by randomly moving the pressure probe over the cross section to see if there are any significant variations, i.e., greater than about 100 mm H_2O (4 in. H_2O). If there are significant variations, check the location for disturbances. If none are found, measure and record the static pressure at each traverse point.

PLANT _____

DATE _____ RUN NO. _____

STACK DIAMETER OR DIMENSIONS, m(in.) _____

BAROMETRIC PRESSURE, mm Hg (in. Hg) _____

CROSS SECTIONAL AREA, $m^2(ft^2)$ _____

OPERATORS _____

PITOT TUBE I.D. NO. _____

AVG. COEFFICIENT, C_p = _____

LAST DATE CALIBRATED _____

SCHEMATIC OF STACK CROSS SECTION

Traverse Pt. No.	Vel. Hd., Δp mm (in.) H_2O	Stack Temperature		P_g[a] mm Hg (in.Hg)	$\sqrt{\Delta p}$
		t_s, °C (°F)	T_s, °K (°R)		
		Average			

[a]If preliminary investigation shows that P_g varies no more than 100 mm H_2O (4 in. H_2O), record one reading.

Figure 2-3. Velocity traverse data.

3.4 Determine the atmospheric pressure.

3.5 Determine the dry stack gas molecular weight. For combustion processes, use Method 3. For processes emitting essentially air, an analysis need not be conducted: use a molecular weight of 29. For other processes, consult the Administrator.

3.6 Obtain the moisture content from Method 5 or by using Preference Method 4.

3.7 Determine the cross sectional area of the stack or duct at the sampling location. Whenever possible, it is better to physically measure the stack dimensions rather than using blueprints.

4. *Calibration.*

4.1 Pitot tube.

4.1.1 Calibration set-up—Calibration shall be done in a flow system having the following essential design features:

4.1.1.1 The flowing gas stream must be confined to a definite cross-sectional area, either circular or rectangular. For circular cross-sections, the minimum duct diameter shall be 30.5 cm (12 inches); for rectangular cross-sections, the width (shorter side) shall be at least 25.4 cm (10 inches).

4.1.1.2 The cross sectional area must be constant over a distance of 10 or more duct diameters. For a rectangular cross section, use an equivalent diameter calculated from he following equation to determine the number of duct diameters:

$$D_e = \frac{2LW}{(L+W)} \quad \text{Equation 2-1}$$

where:

D_e = Equivalent diameter

L = Length

W = Width

To ensure the presence of stable, fully developed flow patterns at the calibration site, or "test section," the site must be located at least 8 diameters downstream and two diameters upstream from the nearest disturbances.

NOTE.—Wind tunnels with well-developed flow patterns (i.e., flow parallel to the duct axis) may also be used.

4.1.1.3 The flow system shall have the capacity to generate a test-section velocity around 915 m/min. (3000 ft/min.), which is the approximate midpoint of the "normal working range" 305 to 1525 m/min. or ~1000 to 5000 ft/min. This velocity must be constant with time, to guarantee steady flow during calibration.

Note that Type-S pitot tube coefficients obtained by single-velocity calibration at the midpoint of the normal working range will generally be valid to within ±3 percent over the entire range. If a more precise correlation between C_p and velocity is desired, the flow system shall have the capacity to generate a number of distinct, time-invariant test-section velocities, covering the normal working range, and calibration data shall be taken at regular velocity intervals between 305 and 1525 m/min. (1000 and 5000 ft/min.). (See Reference 6.9 for details.)

4.1.1.4 Two entry ports, one each for the standard and Type S pitot tubes, shall be cut in the test section; the standard pitot entry port shall be located slightly downstream of the Type S port, so that the standard and Type S impact openings will lie in the same cross-section plane during calibration. To facilitate alignment of the pitot tubes during calibration, it is advisable that the test section be constructed of plexiglas or some other transparent material.

4.1.2 Calibration procedure. Note that this procedure is a general one, and must not be used without first referring to the specific considerations presented in sections 4.1.4–4.1.5. Note also that this procedure applies only to single-velocity calibration; see Preference 6.9 for more details. It is recommended that an identification number be assigned to the pitot tube, and that this number be permanently marked or engraved on the body of the tube; also, one leg of the tube should be marked "A", and the other, "B". To obtain calibration data for both the "A" and "B" sides, proceed as follows:

4.1.2.1 Make sure that the manometer is properly filled and that the oil is free from contamination. Inspect and leak-check all pitot lines; repair or replace if necessary.

4.1.2.2 Level and zero the manometer. Turn on the fan and allow the flow to stabilize. Seal the Type S entry port.

4.1.2.3 Ensure that the manometer is level and zeroed. Position the standard pitot tube at the calibration point (determined as outlined in sections 4.1.4 and 4.1.5), and align it so that its tip is pointed directly into the flow. Particular care should be taken in aligning the tube, to avoid yaw and pitch angles. Make sure that the entry port surrounding the tube is properly sealed.

4.1.2.4 Read ΔP_{std} and record its value in a data table, similar to the one shown in Figure 2-4. Remove the standard pitot tube from the duct and disconnect it from the manometer. Seal the standard entry port.

4.1.2.5 Connect the Type S pitot tube to the manometer. Open the Type S entry port. Check the manometer level and zero. Insert and align the Type S pitot tube so that its "A" side impact opening is at the same point as was the standard pitot tube, and is pointed directly into the flow. Make sure that the entry port surrounding the tube is properly sealed.

4.1.2.6 Read ΔP_s and enter its value in the data table. Remove the Type S pitot tube from the duct and disconnect it from the manometer.

4.1.2.7 Repeat steps 4.1.2.3 through 4.1.2.6 above, until three sets of velocity head readings have been obtained.

4.1.2.8 Repeat steps 4.1.2.3 through 4.1.2.7 above for the B-side of the Type S pitot tube.

PITOT TUBE IDENTIFICATION NUMBER:_____ DATE:_____

CALIBRATED BY:_____

	"A" SIDE CALIBRATION			
RUN NO.	Δp_{std} cm H_2O (in. H_2O)	$\Delta p_{(s)}$ cm H_2O (in. H_2O)	$C_{p(S)}$	DEV.
1				
2				
3				
		AVERAGE		

	"B" SIDE CALIBRATION"			
RUN NO.	Δp_{std} cm H_2O (in. H_2O)	$\Delta p_{(s)}$ cm H_2O (in. H_2O)	$C_{p(S)}$	DEV.
1				
2				
3				
		AVERAGE		

DEV. = $\frac{C_{p(S)} - C_{p(S)(avg.)}}{C_p}$ (MUST BE ≤ 0.01)

DIFFERENCE: $A_{avg} - B_{avg}$ =_____ (MUST BE ≤ 0.01)

Figure 2-4. Pitot tube calibration data.

4.1.3 Calculations.

4.1.3.1 For each of the 6 pairs of velocity head readings (i.e., 3 from Side A and 3 from side B) obtained in section 4.1.2 above, calculate the value of the Type S pitot tube coefficient, as follows:

$$C_{p(S)} = C_{p(std)} \sqrt{\frac{\Delta p_{std}}{\Delta p_S}} \qquad \text{Equation 2-2}$$

where:

$C_{p(S)}$ = Type S pitot tube coefficient
$C_{p(std)}$ = Standard pitot tube coefficient; use 0.99 if the coefficient is unknown and the tuele is designed according to the guidelines in section 2.7
Δp_{std} = Velocity head measured by the standard pitot tube, cm H_2O (in. H_2O)
Δp_S = Velocity head measured by the Type S pitot tube, cm H_2O (in. H_2O)

4.1.3.2 Calculate C_p (side A), the mean A-side coefficient, and C_p (side B), the mean B-side coefficient; calculate the difference between these two average values.

4.1.3.3 Calculate the deviation of each of the three A-side values of $C_{p(S)}$ from $\overline{C}_p$ (side A), and the deviation of each B-side value of $C_{p(S)}$ from $\overline{C}_p$ (side B). Use the following equation:

$$\text{Deviation} = C_{p(S)} - \overline{C}_p(\text{A or B}) \qquad \text{Equation 2-3}$$

4.1.3.4 Calculate σ, the average deviation from the mean, for both the A and B sides of the pitot tube. Use the following equation:

$$\sigma(\text{side A or B}) = \frac{\sum_1^3 |C_{p(S)} - \overline{C}_p(\text{A or B})|}{3} \qquad \text{Equation 2-4}$$

4.1.4 Specific considerations pertaining to calibration of isolated type S pitot tubes. When an isolated Type S pitot tube is to be calibrated, select a calibration point at or near the center of the duct, and follow the procedures outlined in sections 4.1.2 and 4.1.3 above. The coefficients so obtained, i.e., $\overline{C}_p$ (side A) and $\overline{C}_p$ (side B), will be valid for the measurement of stack gas velocities between 305 and 1525 m/min. (1000 and 5000 ft/min.), so long as the isolated pitot tube is used. If, however, the pitot tube is used as a component of a pitobe assembly, the isolated coefficient values may or may not apply; this is discussed more fully in section 4.1.5.

4.1.5 Pitobe Assemblies. Generally, when a Type S pitot tube is used as a component of a pitobe assembly, its A and B-side coefficients will differ appreciably from their respective isolated values if there is aerodynamic interactions among the assembly components. The isolated and assembly coefficient values will only be the same if the assembly is constructed according to the following specifications:

(a) To minimize aerodynamic interactions between the pitot tube and sampling nozzle there must be a separation distance (free-space) of at least 1.90 cm (¾ in.) between the nozzle and pitot tube, with the largest size nozzle (usually 1.3 cm or ½ in., I.d.) in place. (See Figure 2-5.)

(b) To minimize aerodynamic interactions between the thermocouple and pitot tube, the thermocouple wire must be mounted on the pitot tube in such a way that the tip of the wire is in line with, but at least 1.90 cm (¾ in.) from the center of the pitot tube impact openings. (See Figure 2-6.)

(c) To eliminate pitot tube-probe sheath interference, there must be at least 7.62 cm (3 in.) between the leading edge of the probe and the center of the pitot tube impact openings. (See Figure 2-7.)

For those assemblies which either (1) meet requirements (a) through (c) above but have unknown isolated coefficients, or (2) fail to meet these requirements, use the procedures to calibrate the pitot tube-nozzle-thermocouple assemblies outlined in sections 4.1.2 and 4.1.3, in conjunction with the following special considerations, to determine the A and B-side coefficients of the Type S pitot tube:

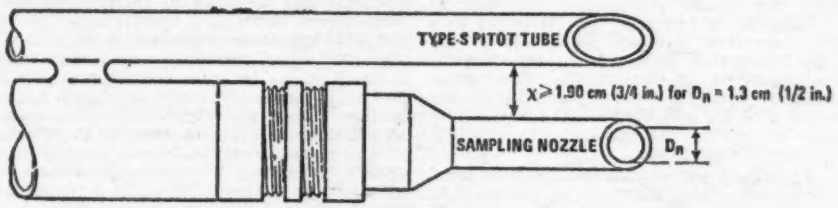


Figure 2-5. Minimum pitot-nozzle separation needed to prevent interference.

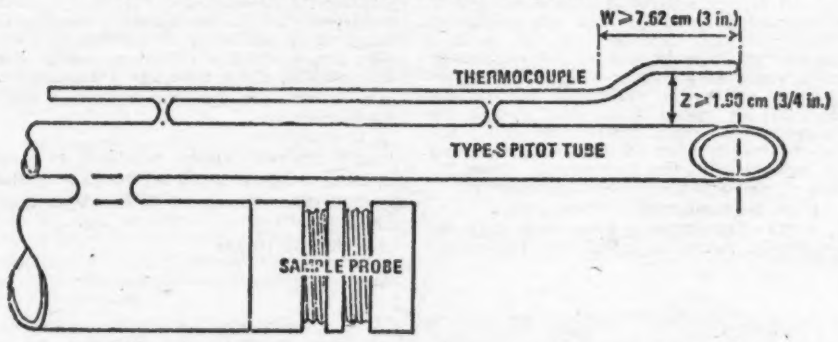


Figure 2-6. Proper thermocouple placement to prevent interference.

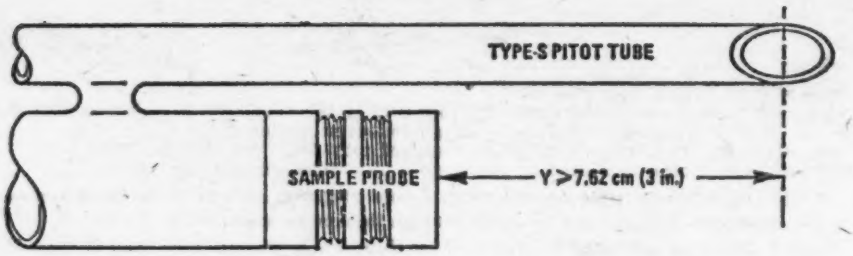
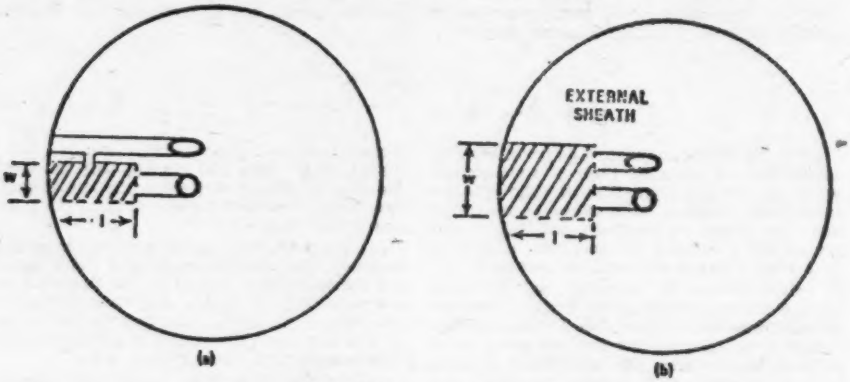


Figure 2-7. Minimum pitot-sample probe separation needed to prevent interference.



$$\text{ESTIMATED SHEATH BLOCKAGE (\%)} = \left[\frac{l \times w}{\text{DUCT AREA}}\right] \times 100$$

Figure 2-8. Projected-area models for typical pitobe assemblies.

(i) Although it is preferable that the calibration point be located at or near the center of the duct, insertion of a probe sheath into a small duct may cause significant cross-sectional area blockade, and yield incorrect coefficient values. Therefore, to minimize the blockage effect, the calibration point may be a few inches off-center if necessary. To keep the actual reduction in C_p due to blockage below 1 percent, it is necessary that the theoretical blockage, as determined by a projected-area model of the probe sheath, be 2 percent or less of the duct cross-sectional area for assemblies without external sheaths (see Figure 2–8a) and 3 percent or less for assemblies with external sheaths (Figure 2–8b).

(ii) For pitobe assemblies in which pitot tube-nozzle interference is a factor (i.e., those in which the pitot-nozzle separation distance is less than 1.90 cm (¾ in.) with a 1.3 cm (½ in.) nozzle in place) the value of C_p will depend somewhat on the amount of free space between the tube and nozzle; in these instances, separate calibrations shall be performed with each of the commonly used nozzle sizes in place. Note that single-velocity calibration technique will be acceptable for this purpose, even though the larger nozzle sizes (>0.635 cm or ¼ in.) are not ordinarily used for isokinetic sampling at velocities around 915 m/min. (3000 ft/min.), which is the calibration velocity.

4.1.6 Recalibration and Field Use.

4.1.6.1 The Type S pitot tube shall be calibrated before its initial use. Thereafter, if the tube has been significantly damaged by field use (for example, if the impact openings are bent out of shape, cut, nicked, or noticeably misaligned), it shall be repaired if possible and recalibrated, or replaced, if necessary.

4.1.6.2 When the Type S pitot tube is used in the field, the appropriate A or B-side coefficient shall be used to perform velocity calculations, depending upon which side of the pitot tube is pointed toward the flow.

4.1.6.3 When sampling a small duct (~12–36 inches in diameter) with a pitobe assembly, the probe sheath can block a significant part of the duct cross-section, causing a reduction in the value of C_p. Therefore, in certain instances it may be necessary, prior to sampling, to make adjustments in the coefficient values obtained by calibration. Consult Reference 6.9 for details.

4.2 Temperature gauges. Calibrate dial and liquid filled bulb thermometers and thermocouple-potentiometer systems against mercury-in-glass thermometers. Ice bath and boiling water (corrected for barometric pressure) are acceptable reference points. For other devices, check with the Administator.

4.3 Barometers. Calibrate against a mercury barometer.

5. *Calculations.*

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

5.1 Nomenclature.

A = Cross sectional area of stack, m^2 (ft^2)
B_{ws} = Water vapor in the gas stream (from Method 5 or Reference Method 4), proportion by volume
C_p = Pitot tube coefficient, dimensionless
K_p = Pitot tube constant,

$$34.97 \frac{m}{sec}\left[\frac{(g/g\text{-mole})(mm\ Hg)}{(°K)(mm\ H_2O)}\right]^{1/2}$$

for the metric system and

$$85.48 \frac{ft}{sec}\left[\frac{(lb/lb\text{-mole})(in.\ Hg)}{(°R)(in.\ H_2O)}\right]^{1/2}$$

for the English system
M_d = Molecular weight of stack gas, dry basis (from Method 3 or other approved methods), g/g-mole (lb/lb-mole)
M_s = Molecular weight of stack gas, wet basis, g/g-mole (lb/lb-mole)
$= M_d(1-B_{ws})+18B_{ws}$ Equation 2–5
P_{bar} = Atmospheric pressure, mm Hg (in. Hg)
P_g = Stack static pressure, mm Hg (in. Hg)
P_s = Absolute stack gas pressure, mm Hg (in. Hg)
$= P_{bar}+P_g$ Equation 2–6
P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg)
Q_{sd} = Dry volumetric stack gas flow rate corrected to standard conditions, dscm/hr (dscf/hr)
t_s = Stack temperature, °C (°F)
T_s = Absolute stack temperature, °K (°R)
$= 273+t_s$ for metric Equation 2–7
$= 460+t_s$ for English Equation 2–8
T_{std} = Standard absolute temperature, 293°K (528°R)
v_s = Average stack gas velocity, m/sec (ft/sec)
Δ_p = Velocity head of stack gas, mm H_2O (in. H_2O)
3600 = Conversion factor, sec/hr
18 = Molecular weight of water, g/g-mole (lb/lb-mole)

5.2 Average stack gas velocity.

$$v_s = K_p C_p (\sqrt{\Delta p})_{avg}\sqrt{\frac{(T_s)_{avg}}{P_s M_s}} \qquad \text{Equation 2–9}$$

NOTE.—Equation 2–7 assumes that T_s, P_s, and M_s do not change appreciably (i.e. >1%) with cross section and with time. If they do, consult with the Administrator to determine an acceptable procedure.

5.3 Average stack gas dry volumetric flow rate.

$$Q_{sd} = 3600(1-B_{ws})v_s A\left[\frac{T_{std}}{T_{s(avg)}}\right]\left[\frac{P_s}{P_{std}}\right] \qquad \text{Equation 2–10}$$

6. *References.*

6.1 Mark, L. S., Mechanical Engineer's Handbook, McGraw-Hill Book Co., Inc., New York, N.Y. 1951.

6.2 Perry, J. H., Chemical Engineers' Handbook, McGraw-Hill Book Co., Inc., New York, N.Y., 1960.

6.3 Shigehara, R. T., W. F. Todd, and W. S. Smith, Significance of Errors in Stack Sampling Measurements. Paper presented at the Annual Meeting of the Air Pollution Control Association, St. Louis, Mo., June 14–19, 1970.

6.4 Standard Method for Sampling Stacks for Particulate Matter. In: 1971 Book of ASTM Standards, Part 23, Philadelphia, Pa., 1971. ASTM Designation D–2928–71.

6.5 Vennard, J. K., Elementary Fluid Mechanics, John Wiley & Sons, Inc., New York, N.Y., 1947.

6.6 ASME, Fluid Meters—Their Theory and Application, ASME, N.Y., 1959.

6.7 ASHRAE Handbook of Fundamentals, 1972, p. 208.

6.8 ASTM Annual Book of ASTM Standards, Part 26, 1974, p. 648.

6.9 Vollaro, R. F., Guidelines for Type-S Pitot Tube Calibration. Paper presented at 1st Annual Meeting, Source Evaluation Society, Dayton, Ohio, September 18, 1975.

METHOD 3—GAS ANALYSIS FOR CARBON DIOXIDE, OXYGEN, EXCESS AIR, AND DRY MOLECULAR WEIGHT

1. *Principle and Applicability.*

1.1 Principle. An integrated or grab gas sample is extracted from a stack and analyzed for percent carbon dioxide and percent oxygen using an Orsat analyzer or, for molecular weight determinations, a Fyrite[1] type combustion gas analyzer.

1.2 Applicability. This method is applicable for determining carbon dioxide and oxygen concentrations, and molecular weight of a sample from a gas stream.

2. *Apparatus.*

Any apparatus which has been demonstrated to yield results acceptable to the Administrator will be considered acceptable for the purposes of this method.

2.1 Grab sample (Figure 3–1).

2.1.1 Probe—Stainless steel or borosilicate glass equipped with a filter (either in-stack or out-stack) to remove particulate matter.

2.1.2 Pump—One-way squeeze bulb, or equivalent, to transport gas sample to analyzer.

2.2 Integrated sample (Figure 3–2).

2.2.1 Probe—Stainless steel or borosilicate glass equipped with a filter (either in-stack or out-stack) to remove particulate matter.

[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

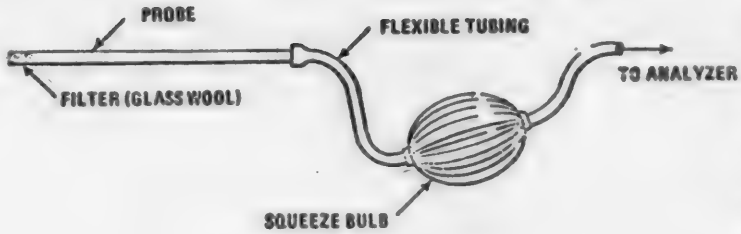


Figure 3-1. Grab-sampling train.

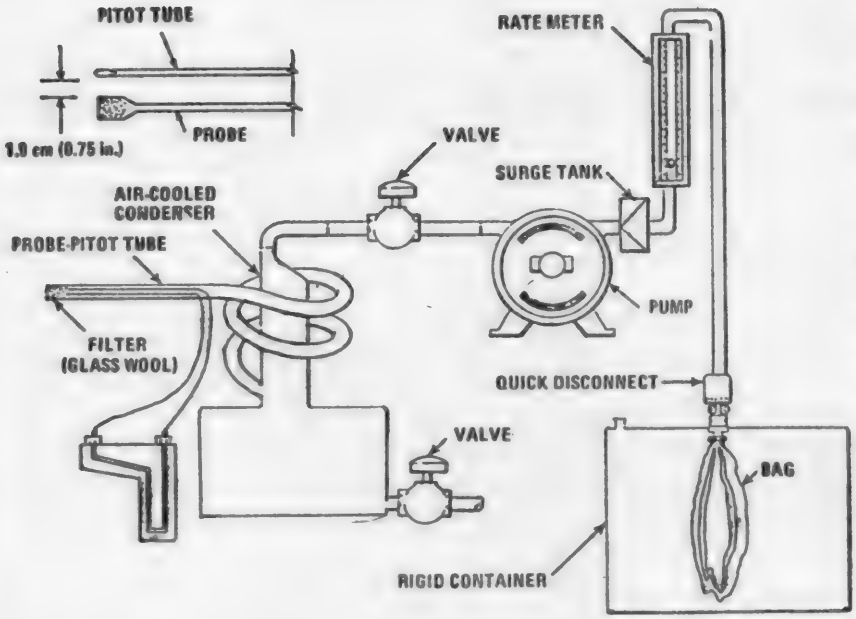


Figure 3-2. Integrated gas-sampling train.

2.2.2 Condenser—Air-cooled condenser, or equivalent, to remove excess moisture.

2.2.3 Valve—Needle valve, to adjust sample gas flow rate.

2.2.4 Pump—Leak-free, diaphragm type, or equivalent, to transport sample gas to the flexible bag. Install a small surge tank between the pump and rate meter to eliminate pulsation effect of diaphragm pump on the rotameter.

2.2.5 Rate meter—Potameter, capable of measuring a flow range from 0 to 1.0 litre per minute.

2.2.6 Flexible bag—Tedlar,[1] or equivalent, with a capacity in the range of 55 to 90 liters. Before each field test run make sure the bag is leak-free by checking it for leaks. To leak check, connect a water manometer and pressurize the bag to 5–10 cm H_2O (2–4 in. H_2O). Allow stand for 10 minutes. Any displacement in the water manometer indicates a leak.

NOTE.—An alternative leak check method is to pressurize the bag to 5–10 cm H_2O or 2–4 in. H_2O and allow to stand overnight. A deflated bag indicates a leak.

2.2.7 Pitot tube—Type S, or equivalent, attached to the probe to allow constant monitoring of the stack gas velocity so that the sampling flow rate can be regulated proportional to the stack gas velocity. The tips of the probe and pitot tube shall be adjacent to each other and the free space between them shall be about 1.9 cm (0.75 in.). When used with this method, the pitot tube need not be calibrated.

2.2.8 Differential pressure gauge—Inclined manometer capable of measuring velocity head to within 10% of the minimum measured value or ±0.013 mm (0.0005 in.), whichever is greater. Below a differential pressure of 1.3 mm (0.05 in.) water gauge, micromanometers with sensitivities of 0.013 mm (0.0005 in.) should be used. However, micromanometers may not easily be adaptable to the existing field conditions and are not easy to use with pulsating flow. Thus, alternative methods or other devices acceptable to the Administrator may be used when conditions warrant.

2.2.9 Manometer—About 28 cm (12 in.). water-filled U-tube manometer, or equivalent, to be used for the flexible bag leak check.

2.2.10 Vacuum gauge—At least 760 mm Hg (30 in. Hg) gauge, to be used for the sampling train leak check.

2.3 Analysis.

2.3.1 Orsat analyzer or Fyrite type combustion gas analyzer. The latter is used only for molecular weight determination. For low CO_2 (less than 4 percent) or high O_2 (greater than 15 percent) concentrations, the measuring burette of the Orsat must have at least 0.01% subdivisions.

3. *Sampling Procedure.*

3.1 Grab sampling. This procedure is primarily used for, but not limited to, determining molecular weight. Other uses must first be approved by the Administrator.

3.1.1 The sampling point in the duct shall be at the centroid of the cross section or at a point no closer to the walls than 1 m (3.28 ft), unless otherwise specified by the Administrator.

3.1.2 Set up the equipment as shown in Figure 3–1, making sure all connections are tight and leak-free by following the procedure in Section 4.

3.1.3 Place the probe in the stack at the sampling point and then purge the sampling line. Draw a sample into the analyzer and analyze according to Section 4.

3.2 Integrated sampling (required when the analytical results will be used to calculate a pollutant emission rate correction factor).

3.2.1 Select the sampling location according to Method 1. In addition to the criteria of Method 1, the sampling location shall be at least 2 diameters downstream from any point of air in-leakage. The downstream distance shall be calculated using the linear distance from the point of air in-leakage, and the diameter of the stack at the sampling location.

3.2.2 A minimum of 8 traverse points, selected according to Method 1, shall be used for circular stacks with diameters less than 0.6 m (2 ft.). A minimum of 12 traverse points, selected according to Method 1, shall be used for all other cases, unless otherwise specified in an applicable subpart, or unless specifically approved by the Administrator.

3.2.3 Leak check the flexible bag as in Section 2.2.6. Set up the equipment as shown in Figure 3–2. Just prior to sampling, leak check the train by placing a vacuum gauge at the condenser inlet pulling a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet at the quick disconnect, and then turning off the pump. The vacuum shall remain stable for at least one minute. Evacuate the flexible bag. Connect the probe and place it in the stack and then purge the sampling line. Now, connect the bag and make sure that all connections are tight and leak free.

3.2.4 Sample at a rate proportional (within 20% of constant proportionality, or as specified by the Administrator) to the stack velocity, traversing all sampling points. Record proportional sampling data as shown in Figure 3–3. When analytical results will be used to calculate a pollutant emission rate correction factor, the sampling Must span the length of time the pollutant emission rate is being determined, sampling at each traverse point for an equal length of time. Collect at least 30 liters (1 ft^3) of sample gas.

3.2.5 Obtain and analyze at least one integrated flue gas sample during each pollutant emission rate determination.

4. *Analytical Procedure.*

4.1 Leak check for Orsat analyzer. Moving an Orsat analyzer frequently causes it to leak. Therefore, an Orsat analyzer should be thoroughly leak-checked on-site before the flue gas sample is introduced into it. The suggested procedure for leak-checking an Orsat analyzer is:

4.1.1 Bring the liquid level in each pipette up to the reference mark on the capillary tubing and then close the pipette stopcock.

TIME	TRAVERSE PT.	Δp mm(in.) H_2O	Q 1pm	$R = \frac{Q}{\sqrt{\Delta p}}$	% DEV.[a]
			AVERAGE		

[a] % DEV = $\left(\frac{R - R_{avg}}{R_{avg}}\right) 100$ (MUST BE ≤ 20%)

Figure 3-3. Proportional sampling data.

4.1.2 Raise the leveling bulb sufficiently to bring the confining liquid meniscus onto the graduated portion of the burette and then close the manifold stopcock.

4.1.3 Record the meniscus position.

4.1.4 Observe the meniscus in the burette and the liquid level in the pipette for movement over the next four minutes.

4.1.5 For the Orsat analyzer to pass the leak-check, two conditions must be met:

4.1.5.1 The liquid level in each pipette must not fall below the bottom of the capillary tubing during this four-minute interval.

4.1.5.2 The meniscus in the burette must not change by more than 0.2 ml during this four-minute interval. For the results to be valid the Orsat analyzer must pass this leak test before and after the analysis.

4.1.6 If the analyzer fails the leak-check procedure, all rubber connections and stopcocks should be checked until the cause of the leak is identified. Leaking stopcocks must be disassembled, cleaned and regreased. Leaking rubber connections must be replaced. After the analyzer is reassembled, the leak-check procedure must be repeated.

4.2 Determination of stack gas molecular weight. (Orsat leak check described above is optional). Within eight hours after the sample is taken, analyze it for percent carbon dioxide and percent oxygen using either an Orsat analyzer or a Fyrite type combustion gas analyzer. Determine the percent of the gas that is nitrogen and carbon monoxide by subtracting the sum of the percent carbon dioxide and percent oxygen from 100 percent.

4.2.1 Grab samples—Repeat the sampling and analysis until the molecular weight from each of three consecutive grab samples differs from their means by no more than 0.3 grams/gram mole (0.3 pounds/pound mole).

4.2.2 Integrated samples—Repeat the analysis until the molecular weight for three consecutive analyses differs from their mean by no more than 0.3 gram/gram mole (0.3 pound/pound mole).

4.3 Determination of O_2, CO_2, or excess air for calculating pollutant emission rate correction factors.

NOTE.—The Fyrite type combustion gas analyzers are not acceptable for this purpose, unless otherwise approved by the Administrator. The results may also be used for determining stack gas molecular weight.

4.3.1 Leak check the Orsat analyzer as described in section 4.1. This procedure is mandatory.

4.3.2 Within four hours after the integrated sample is taken, analyze it for percent carbon dioxide and percent oxygen using an Orsat analyzer. To ensure complete absorption of these gases make repeated passes through the absorbing solution until two consecutive readings are the same. Several passes (3–4) should be made between readings. (If constant readings cannot be obtained after three consecutive readings, replace the absorbing solution.) Determine the percent of the gas that is nitrogen and carbon monoxide by subtracting the sum of the percent carbon dioxide and percent oxygen from 100 percent.

This procedure assumes that carbon monoxide concentration is negligible. If appreciable quantities are expected, alternate procedures subject to approval of the Administrator, must be used.

4.3.3 Repeat the analysis on the integrated sample until each of three consecutive analyses for percent carbon dioxide and percent oxygen differ by no more than 0.3 percent by volume when carbon dioxide is greater than 3% and 0.2 percent by volume when carbon dioxide is less than or equal to 3%.

5. *Calculations.*

5.1 Nomenclature:

M_d = Dry molecular weight (gram/gram mole)
%EA = Percent excess air
$\%CO_2$ = Percent carbon dioxide by volume (dry basis)
$\%O_2$ = Percent oxygen by volume (dry basis)
$\%N_2$ = Percent nitrogen by volume (dry basis)
0.264 = Ratio of oxygen to nitrogen in air, v/v
0.28 = Molecular weight of both nitrogen and CO divided by 100
0.32 = Molecular weight of oxygen divided by 100
0.44 = Molecular weight of carbon dioxide divided by 100

5.2 Excess air. Use equation 3–1 to calculate the percent excess air using the three consecutive analyses that meet the requirements of section 4.3.3. Then calculate the average percent excess air.

$$\%EA = \frac{\%O_2(100)}{0.264\ \%N_2 - \%O_2} \qquad \text{Equation 3–1}$$

NOTE.—The equation above assumes that carbon monoxide concentration is negligible. If appreciable carbon monoxide concentrations are expected, consult with the Administrator.

5.3 Dry molecular weight. Use equation 3-2 to calculate the dry molecular weights using data obtained from sections 4.2.1, 4.2.2, or 4.3.2 and 4.3.3, average the results and report to the nearest 0.1 g/g-mole (0.1 lb/lb-mole).

$$M_d = 0.44(\%CO_2) + 0.32(\%O_2) + 0.28(\%N_2 + \%CO) \quad \text{Equation 3-2}$$

5.4 Carbon dioxide concentration calculation. Using the three consecutive carbon dioxide analyses that meet the requirements of section 4.3.3, calculate the average carbon dioxide concentration.

6. *References.*

6.1 Altshuller, A. P. Storage of Gases and Vapors in Plastic Bags, International Journal of Air and Water Pollution, *6*, 75–81 (1963).

6.2 Connor, William D. and J. S. Nader, Air Sampling with Plastic Bags, Journal of the American Industrial Hygiene Association, *25*, 291–297 (1964).

6.3 "Burrell Manual for Gas Analysts," Seventh edition (1951), Available from Burrell Corporation, 2223 Fifth Avenue, Pittsburgh, Penna. 15219.

METHOD 4—DETERMINATION OF MOISTURE IN STACK GASES

1. *Principle and Applicability.*

1.1 Principle. A gas sample is extracted proportionally from the source and moisture is removed from the gas stream, condensed, and determined either volumetrically or gravimetrically.

1.2 Applicability. This method is applicable for the determination of moisture in stack gas.

Two methods are given. One is a reference method for the accurate determination of moisture content as needed to calculate emission data. The other is an approximation method for moisture content to be subsequently used for setting isokinetic sampling rates. For this latter purpose, the tester may use any alternate means for approximating the moisture content, e.g. drying tubes, wet bulb-dry bulb techniqus, condensation techniques, stoichiometric calculations, previous experience, etc. However, the actual isokinetic rate maintained during a pollutant sampling run and the moisture content used to calculate emission data will not be based on the results of the approximation method (see exception in note below), but will be determined from the data of the reference method, which is normally conducted simultaneously with a pollutant measurement run.

NOTE.—Any of the approximation methods which are shown to the satisfaction of the Administration of yielding results to within 1% H_2O of the reference method results may be used in lieu of the reference method.

These methods are not applicable to gas streams that contain liquid droplets. For these cases, assume that the gas stream is saturated. Determine the average stack gas temperature using gauges described in Method 2 and by traversing according to Method 1. Then obtain the moisture percentage by (1) using a psychometric chart and making appropriate corrections, if stack pressure is different from that of the chart, for absolute pressure or (2) by using saturation vapor pressure tables.

2. *Reference Method.*

The procedure for determining moisture content described in Method 5 is acceptable as a reference method.

2.1 Apparatus. A schematic of the sampling train used in this reference method is shown in Figure 4-1. All components shall be maintained and calibrated according to the procedure outlined in Method 5.

2.1.1 Probe—Stainless steel or glass tubing, sufficiently heated to prevent water condensation and equipped with a filter (either in-stack or heated out-stack) to remove particulate matter.

2.1.2 Condenser—Any system that cools the sample gas stream and allows measurement of the water condensed and moisture leaving the condenser, each to within 1 ml or 1 g. Acceptable means are to measure the condensed water either gravimetrically or volumetrically and to measure the moisture leaving the condenser by (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law or (2) by passing the sample gas stream through a tared silica gel trap with exit gases kept below 20° C (68° F) and determining the weight gain.

2.1.3 Cooling system—Ice bath container and crushed ice, or equivalent, to aid in condensing moisture.

2.1.4 Drying tube—Tube packed with 6–16 mesh indicating-type silica gel, or equivalent, to dry the sample gas and protect the pump and dry gas meter. This may be an integral part of the condenser system, in which case the tube shall be immersed in the ice bath and a thermometer placed at the outlet for monitoring purposes. If approach (1) of section 2.1.2 is used to measure the moisture leaving the condenser, the temperature and pressure must be monitored before the silica gel tube.

2.1.5 Metering system—Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3° C (5.4° F), dry gas meter with ±2 percent accuracy, and related equipment, or other metering systems approved by the Administrator, as required to maintain a proportional sampling rate and to determine sample gas volume.

2.1.6 Barometer—Mercury, aneroid, or other barometers capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby weather bureau station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and the sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

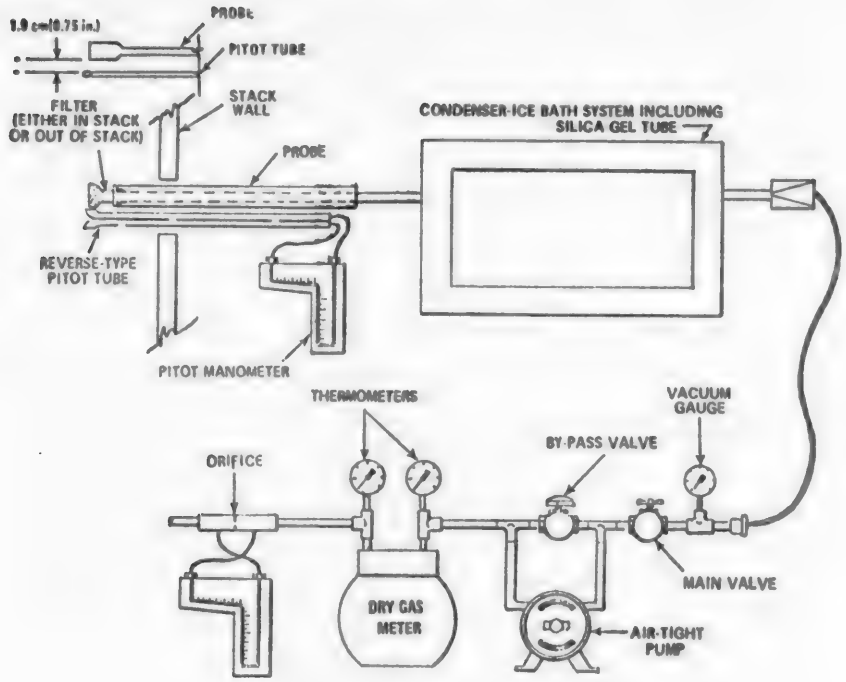


Figure 4-1. Moisture sampling train-reference method.

PLANT _____
LOCATION _____
OPERATOR _____
DATE _____
RUN NO. _____
AMBIENT TEMPERATURE _____
BAROMETRIC PRESSURE _____
PROBE LENGTH m(ft) _____

SCHEMATIC OF STACK CROSS SECTION

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	STACK TEMPERATURE °C (°F)	VELOCITY HEAD (ΔP_s), mm(in.)H_2O	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER (ΔH), mm(in.)H_2O	GAS SAMPLE VOLUME m^3 (ft^3)	GAS SAMPLE TEMPERATURE AT DRY GAS METER		TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °C (°F)
						INLET ($T_{m_{in}}$) °C (°F)	OUTLET ($T_{m_{out}}$) °C (°F)	
TOTAL						Avg.	Avg.	
AVERAGE						Avg.		

Figure 4-2. Field moisture determination-reference method.

	IMPINGER VOLUME, ml	SILICA GEL WEIGHT, g
FINAL		
INITIAL		
DIFFERENCE		

Figure 4-3. Analytical data-reference method.

2.1.7 Pitot tube—Type S, or equivalent, attached to probe to allow constant monitoring of the stack gas velocity so that the sampling flow rate can be regulated proportional to the stack gas velocity. The tips of the probe and pitot tube shall be adjacent to each other and the free space between them shall be about 1.9 cm (0.75 in.). When used with this method, the pitot tube need not be calibrated.

2.1.8 Differential pressure guage—Inclined manometer capable of measuring velocity head to within 10 percent of the minimum measured value or ±0.013 mm (0.0005 in.), in whichever is greater. Below a differential pressure of 1.3 mm (0.05 in.) water gauge, micromanometers with sensitivities of 0.013 mm (0.0005 in.) should be used. However, micromanometers are not easily adaptable to field conditions and are not easy to use with the pulsating flow. Thus, methods or other devices acceptable to the Administrator may be used when conditions warrant.

2.1.9 Temperature gauge—Thermocouple, liquid filled bulb thermometer, bimetallic thermometer, mercury-in-glass thermometer, or other gauges that are capable of measuring temperature to within 1.5 percent of the minimum absolute stack temperature.

2.1.10 Graduated cylinder and/or balance—To measure condensed water and moisture caught in the silica gel to within 1 ml or 1 g. Graduated cylinders shall have subdivisions no greater than 2 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. These balances are suitable for use here.

2.1.11 Temperature and pressure gauges—If Dalton's law is used to monitor temperature and pressure at condenser outlet. The temperature gauge shall have an accuracy of 1° C (2° F). The pressure gauge shall be capable of measuring pressure to within 2.5 mm Hg (0.1 in. Hg).

2.1.12 Silica gel—If used to measure moisture leaving condenser, indicating type, 6–16 mesh. If previously used, dry at 175° C (350° F) for 2 hours. New silica gel may be used as received.

2.2 Procedure. The procedure below is written for a condenser system incorporating silica gel and gravimetric analysis to measure the moisture leaving the condenser and volumetric analysis to measure the condensed moisture.

2.2.1 Select the sampling site and minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the range of velocity head using Method 2 for the purpose of making proportional sampling rate calculations. Select a suitable velocity head to correspond to about 0.014 m³/min (0.5 cfm). Select a suitable probe and probe length such that all traverse points can be sampled. Consider sampling from opposite sides (four total sampling ports) for large stacks to enable use of shorter probe lengths. Mark probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point. Weigh and record weight of silica gel to the nearest 0.5 g.

2.2.2 Select a suitable total sampling time of no less than 1 hour such that a minimum total gas sample volume of 0.6 m³ (20 ft³) at standard conditions will be collected and the sampling time per traverse point is not less than 2 min., or some greater time interval as specified by the Administrator.

2.2.3 Set up the sampling train as shown in Figure 4–1. Turn on the probe heating system to about 120° C (248° F) so as to prevent water condensation and allow time for temperature to stabilize. Place crushed ice in the ice bath container. Leak check the train by plugging the probe inlet and pulling a 380 mm Hg (15 in. Hg) vacuum. A leakage rate in excess of 4 percent of the average sampling rate or 0.00057 m³/min. (0.02 cfm), which ever is less, is unacceptable.

2.2.4 During the sampling run, maintain a sampling rate within 20 percent, or as specified by the Administrator, of constant proportionality. For each run, record the data required on the example data sheet shown in Figure 4–2. Be sure to record the initial dry gas meter reading. Record the dry gas meter reading at the beginning and end of each sampling time increment, when changes in flow rates are made, and when sampling is halted. Take other data point readings at each sample point at least once during each time increment.

2.2.5 To begin sampling position the probe tip at the first traverse point. Immediately start the pump and adjust the flow to proportional conditions. Traverse the cross section. Add more ice and, if necessary, salt to maintain a temperature of less than 20° C (68° F) at the silica gel outlet to avoid excessive moisture losses.

2.2.6 After collecting the sample, measure the volume increase of the liquid to the nearest 1 ml. Determine the increase in weight of the silica gel tube to the nearest 0.5 g. Record the information (see example data sheet, Figure 4–3) and calculate the moisture percentage.

2.3 Calculations. Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

2.3.1 Nomenclature.

B_{ws} = Proportion by volume
M_w = Molecular weight of water, 18 g/g-mole (18 lb/lb-mole)
P_m = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter, mm Hg (in. Hg)
P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg)
R = Ideal gas constant, 0.06236 (mm hg)(m³)/(g-mole)(°K) for metric units and 21.83 (in. Hg)(ft³)/(lb-mole)/(lb-mole)(°R) for English units
T_m = Absolute temperature at meter, °K (°R)
T_{std} = Absolute temperature, 293° K (528° R)
V_m = Dry gas volume measured by meter, dcm (dcf)
$V_{m(std)}$ = Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm (dscf)
$V_{wc(std)}$ = Volume of water vapor condensed corrected to standard conditions, m³ (ft³)
$V_{wsg(std)}$ = Volume of water vapor collected in silica gel corrected to standard conditions, m³ (ft³)
V_f = Final volume of condenser contents, ml
V_i = Initial volume, if any, of condenser contents, ml
W_f = Final weight of condenser contents, g
W_i = Initial weight of condenser contents, g
ρ_w = Density of water, 1 g/ml (0.00220 lb/ml)

2.3.2 Volume of water vapor condensed.

$$V_{wc(std)} = \frac{(V_f - V_i)\rho_w R T_{std}}{P_{std} M_w} = K(V_f - V_i) \quad \text{Equation 4-1}$$

where:

K = 0.00134 m³/ml for metric units

= 0.0472 ft³/ml for English units

2.3.3 Volume of water vapor collected in silica gel.

$$V_{wsg(std)} = \frac{(W_f - W_i) R T_{std}}{P_{std} M_w} = K(W_f - W_i) \quad \text{Equation 4-2}$$

where:

K = 0.00134 m³/g for metric units

= 0.0472 ft³/g for English units

2.3.4 Gas volume.

$$V_{m(std)} = V_m \frac{(P_m)(T_{std})}{(P_{std})(T_m)} = K \frac{V_m P_m}{T_m} \quad \text{Equation 4-3}$$

where:
K = 0.3855 °K/mm Hg for metric units
= 17.65 °R/in. Hg for English units

2.3.5 Moisture Content:

$$B_{ws} = \frac{V_{wc} + V_{wsg}}{V_{wc} + V_{wsg} + V_{mc}} \quad \text{Equation 4-4}$$

2.3.6 Proportional sampling constant—For each time increment, calculate

$$V_m/\sqrt{\Delta\rho}.$$

Calculate the average. If the value for any time increment falls beyond 20 percent of the average, reject the results and do run over.

3. *Approximation Method.*

The approximation method described below is presented only as a suggested method.

3.1 Apparatus.

3.1.1 Probe—Stainless steel or glass tubing sufficiently heated to prevent water condensation and equipped with a filter (either in-stack or heated out-stack) to remove particulate matter.

3.1.2 Impingers—Two midget impingers, each with 30 ml capacity, or equivalent.

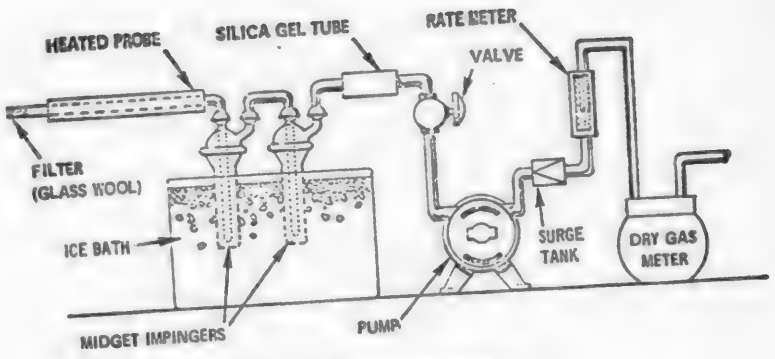


Figure 4-4. Moisture-sampling train.

LOCATION _____ COMMENTS

TEST _____

DATE _____

OPERATOR _____

BAROMETRIC PRESSURE _____

CLOCK TIME	GAS VOLUME THROUGH METER, (Vm), m^3 (ft^3)	RATE METER SETTING m^3/min. (ft^3/min.)	METER TEMPERATURE, °C (°F)

Figure 4-5. Field moisture determination.

3.1.3 Ice bath—Container and ice, to aid in condensing moisture in impingers.

3.1.4 Drying tube—Tube packed with 6–16 mesh indicating-type silica gel, or equivalent, to dry the sample gas and to protect the meter and pump.

3.1.5 Valve—Needle valve, to regulate sample gas flow rate.

3.1.6 Pump—Leak-free, diaphragm type, or equivalent, to pull gas through the train.

3.1.7 Volume meter—Dry gas meter, sufficiently accurate to measure the sample volume within 2 percent, and calibrated over the range of flow rates and conditions actually used during sampling.

3.1.8 Rate meter—Rotameter, to measure the flow range from 0 to 3 lpm (0 to 0.11 cfm).

3.1.9 Graduated cylinder—25 ml.

3.1.10 Barometer—Mercury, aneroid, or other barometers capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby weather bureau station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decreases.

3.1.11 Vacuum gauge—At least 760 mm Hg (30 in. Hg) gauge, to be used for the sampling leak check.

3.2 Procedure.

3.2.1 Place exactly 5 ml distilled water in each impinger. Assemble the apparatus without the probe as shown in Figure 4–4. Leak check by placing a vacuum gauge at the inlet to the first impinger and drawing a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet of the rotameter, and then turning off the pump. The vacuum shall remain constant for a least one minute. Carefully release the vacuum gauge before releasing the rotameter end.

3.2.2 Connect the probe and sample at a constant rate of 2 lpm (0.071 cfm). Continue sampling until the dry gas meter registers about 30 liters (1.1 ft³) or until visible liquid droplets are carried over from the first impinger to the second. Record temperature, pressure, and dry gas meter readings as required by Figure 4–5.

3.2.3 After collecting the sample, combine the contents of the two impingers and measure volume to the nearest 0.5 ml.

3.3 Calculations. The calculation method presented is designed to estimate the moisture in the stack gas and therefore other data, which are only necessary for accurate moisture determinations, are not collected. The following equations adequately estimate the moisture content for the purpose of determining isokinetic sampling rate settings.

3.3.1 Nomenclature.

B_{wm} = Approximate water vapor in the gas stream leaving the impinger, 0.025 proportion by volume

B_{ws} = Water vapor in the gas stream, proportion by volume

M_w = Molecular weight of water, 18 g/g-mole (18 lb/lb-mole)

P_m = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg)

P = Ideal gas constant, 0.06236 (mm Hg)(m³)/(g-mole)(°K) for metric units and 21.83 (in. Hg)(ft³)/(lb-mole)(°R) for English units

T_m = Absolute temperature at meter, °K (°R)

T_{std} = Standard absolute temperature, 293° K (528° R)

V_f = Final volume of impinger contents, ml

V_i = Initial volume of impinger contents, ml

V_m = Dry gas volume measured by dry gas meter, dcm (dcf)

$V_{m(std)}$ = Dry gas volume measured by dry gas meter, corrected to standard conditions, dscm (dscf)

$V_{ws(std)}$ = Volume of water vapor condensed, corrected to standard conditions, m³ (ft³)

ρ_w = Density of water, 1 g/ml (0.00220 lb/ml)

3.3.2 Volume of water vapor collected.

$$V_{ws} = \frac{(V_f - V_i)\rho_w R T_{std}}{P_{std} M_w}$$

$$= K(V_f - V_i) \quad \text{Equation 4-5}$$

Where:

K = 0.00134 m³/ml for metric units

= 0.0472 ft³/ml for English units

3.3.3 Gas volume.

$$V_{m(std)} = V_m \left(\frac{P_m}{P_{std}}\right)\left(\frac{T_{std}}{T_m}\right)$$

$$= K\frac{V_m P_m}{T_m}$$

where:

K = 0.3855 °K/mm Hg for metric units

= 17.65 °R/in. Hg for English units

3.3.4 Approximate moisture content.

$$B_{ws} = \frac{V_{ws}}{V_{ws} + V_{m(std)}} + B_{wm} = \frac{V_{ws}}{V_{ws} + V_{m(std)}} + (0.025) \quad \text{Equation 4-7}$$

4. *Calibration.*

4.1 Use methods and equipment as specified in Methods 2 and 5 and APTD–0576 to calibrate dry gas meter, barometer, and thermometers.

5. *References.*

5.1 Air Pollution Engineering Manual, Danielson, J. A. (ed.), U.S. DHEW, PHS, National Center for Air Pollution Control, Cincinnati, Ohio, PHS Publication No. 999–AP–40, 1967.

5.2 Devorkin, Howard, et al., Air Pollution Source Testing Manual, Air Pollution Control District, Los Angeles, Calif., November 1963.

5.3 Methods for Determination of Velocity, Volume, Dust and Mist Content of Gases, Western Precipitation Division of Joy Manufacturing Co., Los Angeles, Calif., Bulletin WP–50, 1968.

Method 5—Determination of Particulate Emission From Stationary Sources

1. *Principle and Applicability.*

1.1 Principle. Particulate matter is withdrawn isokinetically from the source and collected on glass fiber filter maintained at temperatures in the range of 120±14° C (248±25° F) or such other temperature as specified by an applicable subpart of the standards. The particulate mass is determined gravimetrically after removal of uncombined water.

1.2 Applicability. This method is applicable for the determination of particulate emissions from stationary sources.

2. *Apparatus.*

2.1 Sampling train. A schematic of the sampling train used in this method is shown in Figure 5–1. Commercial models of this train are available. However, if one desires to build his own, complete construction details are described in APTD–0581; for changes from the APTD–0581 document and for allowable modifications to Figure 5–1, see the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD–0576. Since correct usage is important in obtaining valid results, all users should read the APTD–0576 document and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein.

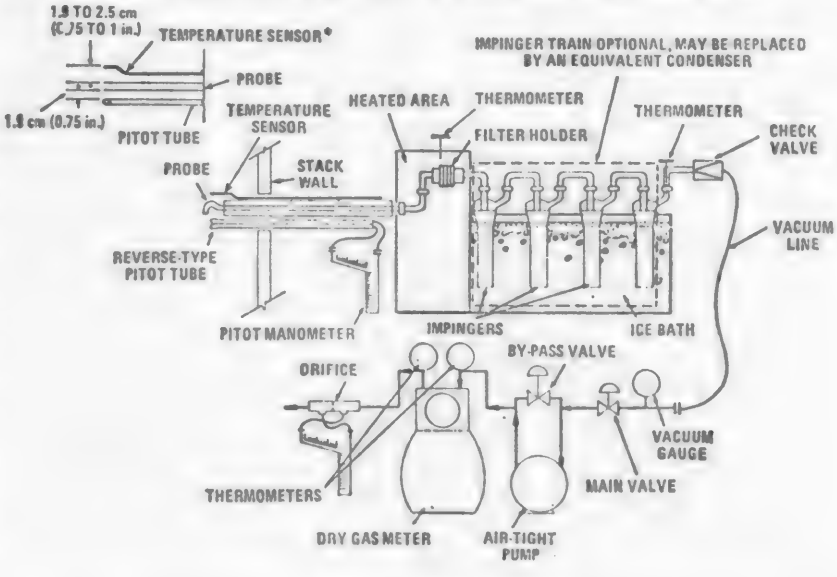


Figure 5-1. Particulate-sampling train.

*If difficulty is expected in inserting the temperature sensor-pitot tube-probe assembly into the stack due to spacing requirements, the temperature sensor may be located between the probe and pitot tube so that the tip of the temperature sensor is no closer than 5cm (2 in.) from the tip of the pitot tube.

2.1.1 Probe nozzle—Stainless steel (316) with sharp, tapered leading edge. The angle of taper shall be≤30° and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise approved by the Administrator. The nozzle shall be constructed from seamless stainless steel tubing. Other configurations and construction material may be used subject to approval from the Administrator.

A range of sizes suitable for isokinetic sampling should be available, e.g., 0.32 cm (⅛ in.) up to 1.27 cm (½ in.) (or larger if higher volume sampling trains are used) inside diameter (ID) nozzles in increments of 0.16 cm (1/16 in.). Each nozzle shall be calibrated according to the procedures outlined in the calibration section.

2.1.2 Probe liner—Borosilicate or quartz glass tubing with a heating system capable of maintaining a gas temperature at the exit end during sampling of no greater than 120±14° C (248±25° F) or no greater than such other temperature as specified by an applicable subpart of the standards. Since the actual temperature at the outlet of the probe is not monitored during sampling, probes constructed according to APTD–0581 and utilizing the calibration curves of APTD–0576 or calibrated according to the procedure outlined in APTD–0576 will be considered as acceptable.

Borosilicate or quartz glass probe liners shall be used for temperatures up to about 480° C (900° F) and quartz liners for temperatures up to about 900° C (1650° F). Both may be used at higher temperatures for short periods of time, but must be approved by the Administrator. The softening temperature for borosilicate is 820° C (1508° F) and for quartz it is 1500° C (2732° F).

When length limitations, i.e. greater than about 2.5 m (8.2 ft), are encountered at temperatures less than 320° C (608° F), stainless steel (316) or Incoloy 825[1] (both of seamless tubing), or other materials as approved by the Administrator, may be used. Metal probes for sampling gas streams at temperatures in excess of 320° C (608° F) must be approved by the Administrator.

2.1.3 Pitot tube—Type S, or other device approved by the Administrator, attached to probe to allow constant monitoring of the stack gas velocity. The face openings of the pitot tube and the probe nozzle shall be adjacent and parallel to each other, not necessarily on the same plane, during sampling. The free space between the nozzle and pitot tube shall be at least 1.9 cm (0.75 in.). The free space shall be set based on a 1.3 cm (0.5 in.) ID nozzle. If the sampling train is designed for sampling at higher flow rates than that described in APTD–0581, thus necessitating the use of larger sized nozzles, the largest sized nozzle shall be used to set the free space.

The pitot tube must also meet the criteria specified in Method 2 and calibrated according to the procedure in the calibration section of that method.

2.1.4 Differential pressure gauge—Inclined manometer capable of measuring velocity head to within 10 percent of the minimum measured value or ±0.013 mm (0.005 in.), whichever is greater. Below a differential pressure of 1.3 mm (0.05 in.) water gauge, micromanometers with sensitivities of 0.013 mm (0.0005 in.) should be used. However, micromanometers are not easily adpatable to field conditions and are not easy to use with pulsating flow. Thus, methods or other devices acceptable to the Administrator may be used when conditions warrant.

2.1.5 Filter holder—Borosilicate glass frit filter support and a silicone rubber gasket. Other materials of construction may be used with approval from the Administrator, e.g., if probe liner is stainless steel, then the filter holder may be stainless steel. The holder design shall provide a positive seal against leakage from the outside or around the filter.

2.1.6 Filter heating system—Any heating system capable of maintaining a temperature around the filter holder during sampling of no greater than 120±14° C (248 ±25° F), or such other temperature as specified by an appplicable subpart of the standards. A temperature gauge capable of measuring temperature to within 30° C (5.4° F) shall be installed such that temperature around the filter holder can be regulated and monitored during sampling. Heating systems other than shown in APTD–0581 may be used.

2.1.7 Condenser—Any system that cools the sample gas stream and allows measurement of the water condenser and moisture leaving the condenser, each to within 1 ml or 1 g. Acceptable means are to measure the condensed water either gravimetrically or volumetrically and to measure the moisture leaving the condenser by (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law or (2) by passing the sample gas stream through a tared silica gel trap with exit gases kept below 20° C (68° F) and determining the weight gain.

NOTE.—If "condensible particulate matter" is desired, in addition to moisture content, the following system shall be used—four impingers connected in series with ground glass, leak free fittings or any similarly leak free noncontaminating fittings. The first, third, and fourth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with a 1.3 cm (½ in.) ID glass tube extending to about 1.3 cm (½ in.) from the bottom of the flask. The second impinger shall be of the Greenburg-Smith design with the standard tip. Individual States or control agencies requiring this information shall be contacted as to the sample recovery and analysis of the impinger contents.

For purposes of writing the procedure of this method, the system described in the note above will be used for determining the moisture content of the stack gas. Modifications (e.g. using flexible connections between the impingers or using materials other than glass) may be used with approval from the Administrator.

If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel still be used between the condenser system and pump to prevent moisture condensation in the pump and metering devices.

Unless otherwise specified by the Administrator, flexible vacuum lines may be used to connect the filter holder to the condenser.

2.1.8 Metering system—Vacuum gauge, leak-free pump, thermometers capable of measuring temperature within 3° C (5.4° F), dry gas meter with 2 percent accuracy, and related equipment, or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume. Sampling trains utilizing metering systems designed for higher flow rates than that described in APTD–0581 or APTD–0576 may be used provided that the specifications in section 2 of this method are met. When the metering system is used in conjunction with a pilot tube, the system shall enable checks of isokinetic rates.

2.1.9 Barometer—Mercury, aneroid, or other barometers capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby weather bureau station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.10 Gas density determination equipment—Temperature and pressure gauges and gas analyzer as described in Methods 2 and 3.

2.1.11 Temperature and pressure gauges—If Dalton's law is used to monitor temperature and pressure at condenser outlet. The temperature gauge shall have an accuracy of 1° C (2° F). The pressure gauge shall be capable of measuring pressure to within 2.5 mm Hg (0.1 in. Hg). If silica gel is used in the condenser system the temperature and pressure must be measured before the silica gel component.

2.2 Sample recovery.

2.2.1 Probe liner and probe nozzle brushes—Nylon bristles with stainless steel wire handles. The probe brush shall have extensions, at least as long as the probe, of stainless steel, nylon, teflon, or similarly inert material. Both brushes shall be properly sized and shaped to brush out the probe liner and nozzle.

2.2.2 Glass wash bottles—Two.

2.2.3 Glass sample storage containers—Chemically resistant, borosilicate glass bottles, for acetone washes, 500 ml or 1,000 ml. Screw cap closures shall be teflon rubber-backed liners or of such construction so as to be leak free and prevent chemical attack from the acetone. (Narrow mouth glass bottles have been found to be less prone to leakage.) Other types of containers must be approved by the Administrator.

2.2.4 Petri dishes—For filter samples, glass or polyethylene, unless otherwise specified by the Administrator.

2.2.5 Graduated cylinder and/or balance—To measure condensed water to within 1 ml or 1 g. Graduated cylinders shall have subdivisions no greater than 2 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. Any of these balances are suitable for use here and in section 2.3.4.

2.2.6 Plastic storage containers—Air tight containers to store silica gel.

2.2.7 Funnel and rubber policeman—To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

2.3 Analysis.

2.3.1 Glass weighing dishes.

2.3.2 Desiccator.

2.3.3 Analytical balance—To measure to within 0.1 mg.

2.3.4 Balance—To measure to within 0.5 g.

2.3.5 Beakers—250 ml.

2.3.6 Hygrometer—To measure the relative humidity of the laboratory environment.

2.3.7 Temperature gauge—To measure the temperature of the laboratory environment.

3. *Reagents.*

3.1 Sampling.

3.1.1 Filters—Glass fiber filters, without organic binder exhibiting at least 99.95 percent efficiency (≤0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM standard method D 2986–71. Test data from the supplier's quality control program is sufficient for this purpose.

[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

3.1.2 Silica gel—Indicating type, 6–16 mesh. If previously used, dry at 175° C (350° F) for 2 hours. New silica gel may be used as received.

3.1.3 Water—When analysis of the material caught in the impingers is required, distilled water shall be used. Run blanks prior to field use to eliminate a high blank on test samples.

3.1.4 Crushed ice.

3.1.5 Stopcock grease—Acetone insoluble, heat stable silicone grease. This is not necessary if screw-on connectors with teflon sleeves, or similar, are used.

3.2 Sample recovery.

3.2.1 Acetone—Reagent grade, ≤0.001 percent residue, in glass bottles. Acetone from metal containers generally has a high residue blank and should not be used. Sometimes, suppliers transfer acetone to glass bottles from metal containers. Thus, acetone blanks shall be run prior to field use and only acetone with low blank values (≤0.001 percent) shall be used.

3.3 Analysis.

3.3.1 Acetone—Same as 3.2.1.

3.3.2 Desiccant—Anhyrdous calcium sulfate, indicating type.

4. *Procedure.*

4.1 Sampling. The sampling shall be conducted by competent personnel experienced with this test procedure.

4.1.1 Pretest preparation. All the components shall be maintained and calibrated according to the procedure described in APTD–0576, unless otherwise specified herein.

Weigh approximately 200–300 g of silica gel in air tight containers to the nearest 0.5 g. Record the total weight, both silica gel and container, on the container. More silica gel may be used but care should be taken during sampling that it is not entrained and carried out from the impinger. As an alternative, the silica gel may be weighed directly in the impinger or its sampling holder just prior to the train assembly.

Check filters visually against light for irregularities and flaws or pinhole leaks. Label a filter of proper diameter on the back side near the edge using numbering machine ink. As an alternative, label the shipping container (glass or plastic petri dishes) and keep the filter in this container at all times except during sampling and weighing.

Desiccate the filters at 20±5.6° C (68±10° F) and ambient pressure for at least 24 hours and weigh at 6 or more hour intervals to a constant weight, i.e., ≤0.5 mg change from previous weighing, and record results to the nearest 0.1 mg. During each weighing the filter must not be exposed to the laboratory atmosphere for a period greater than 2 minutes and a relative humidity above 50 percent.

4.1.2 Preliminary determinations. Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2 and moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate calculations. Estimates may be used. However, final results will be based on actual measurements made during the test.

Select a nozzle size based on the range of velocity heads such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the differential pressure gauge is capable of measuring the minimum velocity head value to within 10 percent, or as specified by the Administrator.

Select a suitable probe liner and probe length such that all traverse points can be sampled. Consider sampling from opposite sides for large stacks to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that the sampling time per point is not less than 2 min. or some greater time interval as specified by the Administrator and the sample volume that will be taken will exceed the required minimum total gas sample volume specified in the test procedures for the specific industry. The latter is based on an approximate average sampling rate. Note also that the minimum total sample volume is corrected to standard conditions.

It is recommended that ½ or an integral number of minutes be sampled at each point in order to avoid timekeeping errors.

In some circumstances, e.g. batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

4.1.3 Preparation of collection train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

Place 100 ml of water in each of the first two impingers, leave the third impinger empty, and place approximately 200–300 g or more, if necessary, of preweighed silica gel in the fourth impinger. Record the weight of the silica gel and container to the nearest 0.5 g. Place the container in a clean place for later use in the sample recovery.

Using a tweezer or clean disposable surgical gloves, place the labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as not to allow the sample gas stream to circumvent the filter. Check filter for tears after assembly is completed.

When glass liners are used, install selected nozzle using a Viton A [1] O-ring when stack temperatures are less than 260° C (500° F) or an asbestos string gasket when temperatures are higher. The Viton A O-ring and asbestos string gasket are installed as a seal where the nozzle is connected to a glass liner. See APTD–0576 for details. When metal liners are used, install the nozzle as above or by a leak free direct mechanical connection. Mark probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Unless otherwise specified by the Administrator, attach a temperature probe to the metal sheath of the sampling probe so that the sensor extends beyond the probe tip and does not touch any metal. Its position should be about 1.9 to 2.54 cm (0.75 to 1 in.) from both the pitot tube and probe nozzle to avoid interference with the gas flow.

Set up the train as in Figure 5–1, using, if necessary, a very light coat of silicone grease on all ground glass joints, greasing only the outer portion (see APTD–0576) to avoid possibility of contamination by the silicone grease. With approval from the Administrator, a glass cyclone may be used between the probe and filter holder.

Place crushed ice around the impingers.

4.1.4 Leak check procedure—After the sampling train has been assembled, turn on and set the filter and probe heating system to the power required to reach a temperature of 120±14° C (248±25° F) or such other

[1] Mention of trade names is not intended to constitute endorsement by EPA.

temperature as specified by an applicable subpart of the standards for the leak check. (If water condensation is not a problem the probe and/or filter heating system need not be used.) Allow time for the temperature to stabilize. If a Viton A O-ring or other leak free connection is used in assembling the probe nozzle to the probe liner, leak check the train at the sampling site by plugging the nozzle and pulling a 380 mm Hg (15 in. Hg) vacuum.

NOTE.—A lower vacuum may be used provided that it is not exceeded during the test.

If an asbestos string is used, do not connect the probe to the train during the leak check. Instead, leak check the train as above by first plugging the inlet to the filter holder. Then connect the probe to the train and leak check at about 25 mm Hg (1 in. Hg) vacuum. A leakage rate in excess of 4 percent of the average sampling rate or 0.00057 m³/min. (0.02 cfm), whichever is less, is unacceptable in either case.

PLANT _____

LOCATION _____

OPERATOR _____

DATE _____

RUN NO. _____

SAMPLE BOX NO. _____

METER BOX NO. _____

METER $\Delta H_@$ _____

C FACTOR _____

PITOT TUBE COEFFICIENT, C_p _____

SCHEMATIC OF STACK CROSS SECTION

AMBIENT TEMPERATURE _____

BAROMETRIC PRESSURE _____

ASSUMED MOISTURE, % _____

PROBE LENGTH, m (ft) _____

NOZZLE IDENTIFICATION NO. _____

AVERAGE CALIBRATED NOZZLE DIAMETER, cm (in.) _____

PROBE HEATER SETTING _____

LEAK RATE, m^3/min (cfm) _____

PROBE LINER MATERIAL _____

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	STATIC PRESSURE mm Hg (in Hg)	STACK TEMPERATURE (T_s) °C (°F)	VELOCITY HEAD (ΔP_s), mm(in.)H_2O	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER mm H_2O (in. H_2O)	GAS SAMPLE VOLUME m^3 (ft^3)	GAS SAMPLE TEMPERATURE AT DRY GAS METER		FILTER HOLDER TEMPERATURE, °C (°F)	TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °C (°F)
							INLET °C (°F)	OUTLET °C (°F)		
TOTAL							Avg.	Avg.		
AVERAGE							Avg.			

Figure 5-2. Particulate field data.

The following leak check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with by-pass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the by-pass valve until 380 mm Hg (15 in. Hg) vacuum is reached. Do not reverse direction of by-pass valve. This will cause water to back up into the filter holder. If 380 mm Hg (15 in. Hg) is exceeded, either leak check at this higher vacuum or end the leak check as shown below and start over.

When the leak check is completed, first slowly remove the plug from the inlet to the probe or filter holder and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward into the filter holder and silica gel from being entrained backward into the third impinger.

Leak checks shall be conducted as described whenever the train is disengaged, e.g. for silica gel or filter changes during the test, prior to each test run, and at the completion of each test run. If leaks are found to be in excess of the acceptable rate, the test will be considered invalid. To reduce lost time due to leakage occurrences, it is recommended that leak checks be conducted between port changes at the highest vacuum reading drawn during that sampling traverse.

4.1.5 Particulate train operation—During the sampling run, isokinetic sampling rate to within 10 percent, or as specified by the Administrator, of true isokinetic and the temperature around the filter of no greater than 120±14° C (248±25° F), or as specified by an applicable subpart of the standards, shall be maintained.

For each run, record the data required on the example data sheet shown in Figure 5-2. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, and when sampling is halted. Take other data point readings at least once at each sample point during each time increment and additional readings when significant changes (20 percent variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer.

Clean the portholes prior to the test run to minimize the chance of sampling the deposited material. To begin sampling, remove the nozzle can, verify that the filter and probe are up to temperature, and that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediate start the pump and adjust the flow to isokinetic conditions. Nomographs are available for sampling trains using type S pitot tubes with 0.85±0.02 coefficient and when sampling in air or a stack gas with equivalent density (molecular weight equal to 29±4), which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations. APTD-0576 details the procedure for using these nomographs. If C_p and M_s are outside the above stated ranges, do not use the nomograph unless appropriate steps (see Peference 7.7) are taken to compensate for the deviations.

When the stack is under significant negative stack pressure (height of impinger stem), take care to close the coarse adjust valve before inserting the probe into the stack to avoid water backing into the filter

holder. If necessary, the pump may be turned on with the coarse adjust valve closed.

When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.

Traverse the stack cross section, as required by Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes, to minimize chance of extracting deposited material.

During the test run, make periodic adjustments to keep the temperature around the filter holder at the proper temperature and add more ice and, if necessary, salt to maintain a temperature of less than 20°C (68°F) at the condenser/silica gel outlet to avoid excessive moisture losses. Also, periodically check the level and zero of the manometer.

If the pressure drop across the filter becomes too high making isokinetic sampling difficult to maintain, the filter may be replaced in the midst of a sample run. It is recommended that another complete filter assembly be used rather than attempting to change the filter itself. After the new filter or filter assembly is installed conduct a leak check. The particulate weight shall include the summation of all filter assembly catches.

A single train shall be used for the entire sample run, except for filter and silica gel changes. However, if approved by the Administrator, two or more trains may be used for a single test run when there are two or more ducts or sampling ports. The results shall be the total of all sampling train catches.

At the end of the sample run, turn off the pump, remove the probe and nozzle from the stack, and record the final dry gas meter reading. Perform a leak check at a vacuum equal to or greater than the maximum reached during sampling. Calculate percent isokinetic (see calculation section) to determine whether another test run should be made. If there is difficulty in maintaining isokinetic rates due to source conditions, consult with the Administrator for possible variance on the isokinetic rates.

4.2 Sample recovery. Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool.

When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to prevent losing or gaining particulate matter. Do not cap off the probe tip tightly while the sampling train is cooling down as this would create a vacuum in the filter holder, thus drawing water from the impingers into the filter.

Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate, if present. Wipe off the silicone grease from the filter inlet where the probe was fastened and cap it. Remove the umbilical cord from the last impinger and cap the impinger. If a flexible line is used between the first impinger or condenser and the filter holder, disconnect the line at the filter holder and let any condensed water or liquid drain into the impingers or condenser. After wiping off the silicone grease, cap off the filter holder outlet and impinger inlet. Either ground glass stoppers or plastic caps or serum caps may be used to close these openings.

Transfer the probe and filter-impinger assembly to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Save a portion of the acetone used for cleanup as a blank. Place about 200 ml of this acetone taken directly from the wash bottle being used in a glass sample container labeled "acetone blank."

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

Container No. 1. Carefully remove the filter from the filter holder and place in its identified petri dish container. Use a pair of tweezers and/or clean disposable surgical gloves to handle the filter. If it is necessary to fold the filter, do so such that the particulate cake is inside the fold. Quantitatively remove any particulate matter and/or filter which adheres to the filter holder gasket by carefully using a dry nylon bristle brush and/or a sharp-edged blade and place into this container. Seal the container.

Container No. 2. Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover particulate matter or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with acetone and placing the wash into a glass container in the following manner:

Distilled water may be used instead of acetone when approved by the Administrator or shall be used when specified by the Administrator. In these cases, save a water blank and follow Administrator's directions on analysis.

Carefully remove the probe nozzle and clean the inside surface by rinsing with acetone from a wash bottle and brushing with a nylon bristle brush. Brush until acetone rinse shows no visible particles, after which make a final rinse of the inside surface with acetone.

Brush and rinse with acetone the inside parts of the Swagelok fitting in a similar way until no visible particles remain.

Rinse the probe liner with acetone by tilting the probe and squirting acetone into its upper end, while rotating the probe so that all inside surfaces will be rinsed with acetone. Let the acetone drain from the lower end into the sample container. A funnel may be used to aid in transferring liquid washes to the container. Follow the acetone rinse with a probe brush. Hold the probe in an inclined position, squirt acetone into the upper end as the probe brush is being pushed with a twisting action through the probe, hold a sample container underneath the lower end of the probe, and catch any acetone and particulate matter which is brushed from the probe. Run the brush through the probe three times or more until no visible particulate matter is carried out with the acetone or remains in the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times since metal probes have small crevices in which particulate matter can be entrapped. Rinse the brush with acetone and quantitatively collect these washings in the sample container. After the brushing make a final acetone rinse of the probe as described above.

It is recommended that two people be used to clean the probe to minimize losing the sample. Between sampling runs, keep brushes clean and protected from contamination.

After ensuring that all joints are wiped clean of silicone grease, clean the inside of the front half of the filter holder by rubbing the surfaces with a nylon bristle brush and rinsing with acetone. Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush and filter holder. After all acetone washings and particulate matter are collected in the sample container, tighten the lid on the sample container so that acetone will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label container to clearly identify its contents.

Container No. 3. Note color of indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the fourth impinger to the original container and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the walls and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure under analysis.

Impinger water. Treat the impingers or condenser as follows: Make a notation of any color or film in the liquid catch. Measure the liquid which is in the first three impingers to within ±1 ml by using a graduated cylinder or, if available, to within ±0.5 g by using a balance. Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas.

If analysis of the impinger catch is not required, discard the liquid after measuring and recording the volume or weight. If analysis of the impinger catch is required, leave the impingers intact to transfer the liquid, cap off the inlet, and pour the liquid through the outlet into the graduated cylinder or into a sample container after its weight has been determined.

If a different type of condenser is used, measure the amount of moisture condensed either volumetrically or gravimetrically.

4.3 Analysis. Record the data required on the example sheet shown in Figure 5–3. Handle each sample container as follows:

Container No. 1. Leave in shipping container or transfer the filter and any loose particulate from the sample container to a tared glass weighing dish and desiccate for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh to a constant weight and report the results to the nearest 0.1 mg. For purposes of this section 4.3, the term "constant weight" means a difference of no more than 0.5 mg or 1 percent of total weight less tare weight, whichever is greater, between two consecutive weighings, with no less than 6 hours of desiccation time between weighings and no more than 2 minutes exposure to the laboratory atmosphere (must be less than 50 percent relative humidity) during weighing.

Plant _____

Date _____

Run No. _____

Relative Humidity _____

Amount liquid lost during transport _____

Acetone blank volume, ml _____

Acetone wash volume, ml _____

Acetone blank concentration, mg/mg (equation 5-4) _____

Acetone wash blank, mg (equation 5-5) _____

CONTAINER NUMBER	WEIGHT OF PARTICULATE COLLECTED, mg		
	FINAL WEIGHT	TARE WEIGHT	WEIGHT GAIN
1			
2			
TOTAL			
		Less acetone blank	
		Weight of particulate matter	

	VOLUME OF LIQUID WATER COLLECTED		
	IMPINGER VOLUME, ml.	SILICA GEL WEIGHT, g	
FINAL			
INITIAL			
LIQUID COLLECTED			
TOTAL VOLUME COLLECTED		g*	ml

CONVERT WEIGHT OF WATER TO VOLUME BY DIVIDING TOTAL WEIGHT INCREASE BY DENSITY OF WATER (1g/ml).

$$\frac{\text{INCREASE, g}}{\text{1 g/ml}} = \text{VOLUME WATER, ml}$$

Figure 5-3. Analytical data.

Container No. 2. Note level of liquid in container and confirm on analysis sheet whether or not leakage occurred during transport. Measure the liquid in this container either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Transfer the contents to a tared 250 ml beaker, and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

Container No. 3. Weigh the spent silica gel to the nearest 0.5 g using a balance. This step may be conducted in the field.

"Acetone Blank" Container. Measure acetone in this container either volumetrically or gravimetrically. Transfer the acetone to a tared 250 ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

5. *Calibration.*

Maintain a laboratory log of all calibrations.

5.1 Probe nozzle. Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in.). Make 3 separate measurements using different diameters each time and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.).

When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use.

Each nozzle shall be permanently and uniquely identified.

5.2 Pitot tube. The pitot tube shall be calibrated according to the procedure outlined in Method 2.

5.3 Dry gas meter and orifice meter. Both meters shall be calibrated according to the procedure outlined in APTD–0576. When a diaphragm pump is used, assure that there is no leak.

5.4 Probe heater calibration. The probe heating system shall be calibrated according to the procedure contained in APTD–0576. Probes constructed according to APTD–0581 need not be calibrated if the calibration curves in APTD–0576 are used.

5.5 Temperature gauges. Calibrate dial and liquid filled bulb thermometers and thermocouple-potentiometer systems against mercury-in-glass thermometers. Ice bath and boiling water (corrected for barometric pressure) are acceptable reference points. For other devices, check with the Administrator.

6. *Calculations.*

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

6.1 Nomenclature

A_n = Cross sectional area of nozzle, m^2 (ft^2)
B_{ws} = Water vapor in the gas stream, proportion by volume
C_a = Acetone blank residue concentration, mg/g
c_s = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, g/dscm (g/dscf)
I = Percent of isokinetic sampling
m_n = Total amount of particulate matter collected, mg
M_w = Molecular weight of water, 18 g/g-mole (18 lb/lb-mole)
m_a = Mass of residue of acetone after evaporation, mg
P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg)
P_s = Absolute stack gas pressure, mm Hg (in. Hg)
P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg)
R = Ideal gas constant, 0.06236 mm Hg-m^3/°K-g-mole (21.83 in. Hg-ft^3/°P-lb-mole)
T_m = Absolute average dry gas meter temperature see (Figure 5–2), °K (°R)
T_s = Absolute average stack gas temperature (see Figure 5–2), °K (°R)
T_{std} = Standard absolute temperature, 293° K (528° R)
V_a = Volume of acetone blank, ml
V_{aw} = Volume of acetone used in wash, ml
V_{lc} = Total volume of liquid collected in impingers and silica gel (see Figure 5-3), ml
V_m = Volume of gas sample as measured by dry gas meter, dcm (dcf)
$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter corrected to standard conditions, dscm (dscf)
$V_{w(std)}$ = Volume of water vapor in the gas sample corrected to standard conditions, scm (scf)
v_s = Stack gas velocity, calculated by Method 2, Equation 2–7 using data obtained from Method 5, m/sec (ft/sec)
W_a = Weight of residue in acetone wash, mg
ΔH = Average pressure differential across the orifice meter (see Figure 5–2), mm H_2O (in. H_2O)
ρ_a = Density of acetone, mg/ml (see label on bottle)
ρ_w = Density of water, 1 g/ml (0.00220 lb/ml)
Θ = Total sampling time, min
13.6 = Specific gravity of mercury
60 = Sec/min
100 = Conversion to percent

6.2 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 5–2).

6.3 Dry gas volume. Correct the sample volume measured by the dry gas meter to standard conditions (20° C, 760 mm Hg or 68° F, 29.92 in. Hg) by using Equation 5–1.

$$V_{m(std)} = V_m\left(\frac{T_{std}}{T_m}\right)\left[\frac{P_{bar} + \frac{\Delta H}{13.6}}{P_{std}}\right] = KV_m\left[\frac{P_{bar} + \Delta H/13.6}{T_m}\right]$$

Equation 5–1

where:

K = 0.3855 °K/mm Hg for metric units

= 17.65 °R/in. Hg for English units

6.4 Volume of water vapor.

$$V_{w(std)} = V_{lc}\left(\frac{\rho_w}{M_w}\right)\left(\frac{RT_{std}}{P_{std}}\right) = KV_{lc}$$

Equation 5–2

where:
K = 0.00134 m³/ml for metric units
= 0.0472 ft³/ml for English units

6.5 Moisture content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}}$$

Equation 5–3

6.6 Acetone blank concentration.

$$C_a = \frac{m_a}{V_a \rho_a}$$

Equation 5–4

6.7 Acetone wash blank.

$$W_a = C_a V_{aw} \rho_a$$

Equation 5–5

6.8 Total particulate weight. Determine the total particulate catch from the sum of the weights obtained from containers 1 and 2 less the acetone blank (see Figure 5–3).

6.9 Particulate concentration.

$$c_s = (0.001\ g/mg)(m_n/V_{m(std)})$$

Equation 5–6

6.10 Conversion factors:

From—	To—	Multiply by—
scf	m³	0.0283
g/ft³	gr/ft³	15.4
g/ft³	lb/ft³	2.205×10^{-3}
g/ft³	g/m³	35.31

6.11 Isokinetic variation.

6.11.1 Calculations from raw data.

$$I = \frac{100T_s[KV_{lc} + (V_m/T_m)P_{bar} + \Delta H/13.6)]}{60\theta v_s P_s A_n}$$

Equation 5–7

where:

K = 0.00346 mm Hg-m³/ml-°K for metric units

= 0.00267 in. Hg-ft³/ml-°R for English units

6.11.2 Calculations from intermediate values.

$$I = \frac{T_s V_{m(std)} P_{std} 100}{T_{std} v_s \theta A_n P_s 60(1 - B_{ws})}$$

$$= K\frac{T_s V_{m(std)}}{P_s v_s A_n \theta (1 - B_{ws})}$$

Equation 5–8

where:

K = 4.323 for metric units

= 0.0944 for English units

6.12 Acceptable results. If 90 percent ≤I ≤110 percent, the results are acceptable. If the results are low in comparison to the standards and I is beyond the acceptable range, the Administrator may option to accept the results. Use reference 7.4 to make judgments. Otherwise, reject the results and repeat the test.

7. *Reference.*

7.1 Addendum to Specifications for Incinerator Testing at Federal Facilities, PHS, NCAPC, Dec. 6, 1967.

7.2 Martin, Robert M., Construction Details of Isokinetic Source Sampling Equipment, Environmental Protection Agency, APTD–0581, April 1971.

7.3 Rom, Jerome J., Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment, Environmental Protection Agency, APTD–0576, March 1972.

7.4 Smith, W. S., R. T. Shigehara, and W. F. Todd. A Method of Interpreting Stack Sampling Data, Paper presented at the 63d Annual Meeting of the Air Pollution Control Association, St. Louis, Mo., June 14–19, 1970.

7.5 Smith, W. S., et al., Stack Gas Sampling Improved and Simplified with New Equipment, APCA paper No. 67–119, 1967.

7.6 Specifications for Incinerator Testing at Federal Facilities, PHS, NCAPC, 1967.

7.7 Shigehara, R. T., Adjustments in the EPA Nomograph for Different Pitot Tube Coefficients and Dry Molecular Weights, Stack Sampling News 2:4–11, Oct. 1974.

METHOD 6—DETERMINATION OF SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and Applicability.*

1.1 Principle. A gas sample is extracted from the sampling point in the stack. The acid mist (including sulfur trioxide) and the sulfur dioxide are separated. The sulfur dioxide fraction is measured by the barium-thorin titration method.

1.2 Applicability. This method is applicable for the determination of sulfur dioxide emissions from stationary sources. The minimum detectable limit of the method has been determined to be 3.4 mg of SO_2/m^3 (2.1×10^{-7} lb/ft³). No upper limit has been established.

2. *Apparatus.*

2.1 Sampling. See Figure 6–1.

2.1.1 Probe—Borosilicate glass, approximately 5 to 6 mm ID, with a heating system to prevent water condensation and equipped with a filter (either in-stack or heated out-stack) to remove particulate matter including sulfuric acid mist.

2.1.2 Bubbler and impingers—One midget bubbler, with medium coarse glass frit and borosilicate or quartz glass wool packed in top (see Figure 6–1) to prevent sulfuric acid mist carryover; and three midget impingers, each with 30 ml capacity, or equivalent. The bubbler and midget impingers shall be connected in series with leak free glass connectors. Silicone grease may be used, if necessary, to prevent leakage.

2.1.3 Glass wool-Borosilicate or quartz.

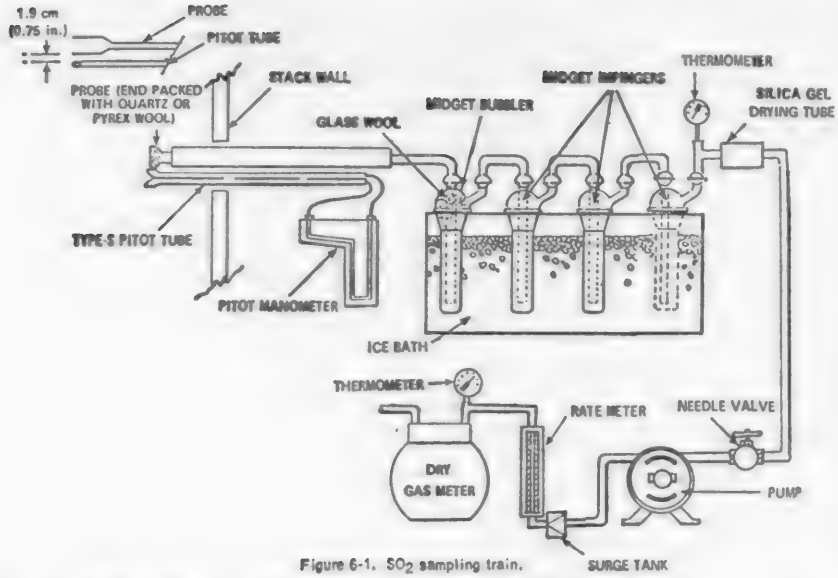


Figure 6-1. SO_2 sampling train.

2.1.4 Stopcock grease—Aceteone insoluble, heat stable silicone grease may be used, if necessary.

2.1.5 Drying tube—Tube packed with 6 to 16 mesh indicating-type silica gel, or equivalent, to dry the gas sample and to protect the meter and pump.

2.1.6 Valve—Needle valve, to regulate sample gas flow rate.

2.1.7 Pump—Leak free diaphragm pump, or equivalent, to pull gas through the train.

2.1.8 Volume meter—Dry gas meter, sufficiently accurate to measure the sample volume within 2 percent, calibrated over the range of flow rates and conditions actually used during sampling and equipped with a temperature gauge (dial thermometer, or equivalent).

2.1.9 Flow Meter—Rotameter, or equivalent, to measure flow range from 0–2 lpm (0 to 5 cfh).

2.1.10 Pitot tube—Type S, or equivalent, attached to probe to allow constant monitoring of the stack gas velocity so that the sampling flow rate can be regulated proportional to the stack gas velocity. The tips of the probe and pitot tube shall be adjacent to each other and the free space between them shall be about 1.9 cm (0.75 in.). When used with this method, the pitot tube need not be calibrated.

The pitot tube shall be equipped with an inclined manometer, or equivalent device, capable of measuring velocity head to within 10 percent of the minimum measured value or ±0.013 mm (0.0005 in.), whichever is greater.

2.1.11 Temperature gauge—Dial thermometer, or equivalent, to measure temperature of gas leaving impinger train to within 1° C (2° F).

2.1.12 Barometer—Mercury, aneroid, or other barometers capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby weather bureau station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.13 Vacuum gauge—At least 760 mm Hg (30 in. Hg) gauge, to be used for the sampling train leak check.

2.2 Sample recovery.

2.2.1 Wash bottles—Polyethylene or glass, 500 ml, two.

2.2.2 Storage bottles—Polyethylene, 100 ml, to store impinger samples (one per sample).

2.3 Analysis.

2.3.1 Pipettes—Volumetric type, 5 ml size, 20 ml size (one per sample), and 25 ml size.

2.3.2 Volumetric flasks—100 ml size (one per sample) and 1000 ml sizes.

2.3.3 Burettes—5 ml and 50 ml sizes.

2.3.4 Erlenmeyer flasks—250 ml size (one for each sample, blank, and standard).

2.3.5 Dropping bottle—125 ml size, to add indicator.

2.3.6—Graduated cylinder—100 ml size.

3. *Reagents.*

Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available; otherwise use best available grade.

3.1 Sampling.

3.1.1 Water—Deionized, distilled to conform to ASTM specification D1193–72, Type 3.

3.1.2 Isopropanol, 80 percent—Mix 80 ml of isopropanol with 20 ml of deionized, distilled water.

3.1.3 Hydrogen peroxide, 3 percent—Dilute 30 percent hydrogen peroxide 1:9 (v/v) with deionized, distilled water (30 ml is needed per sample). Prepare fresh daily.

3.2 Sample recovery.

3.2.1 Water—Deionized, distilled, as in 3.1.1.

3.2.2 Isopropanol, 80 percent—Mix 80 ml of isopropanol with 20 ml of deionized, distilled water.

3.3 Analysis.

3.3.1 Water—Deionized, distilled, as in 3.1.1.

3.3.2 Isopropanol, 100 percent.

3.3.3 Thorin indicator—1-(o-arsonophenylazo)-2-naphtol-3, 6-disulfonic acid, disodium salt, or equivalent. Dissolve 0.20 g in 100 ml of deionized, distilled water.

3.3.4 Barium perchlorate solution, 0.01 N—Dissolve 1.95 g of barium perchlorate trihydrate [$Ba(ClO_4)_2 \cdot 3H_2O$] in 200 ml distilled water and dilute to 1 liter with isopropanol. $BaCl_2 \cdot 2H_2O$ (1.22 g) may also be used. Standardize as in section 5.2.

3.3.5 Sulfuric acid standard, 0.01 N—Purchase or standardize to ±0.0002 N against 0.01 N NaOH which has previously been standardized against potassium acid phthalate (primary standard grade).

4. *Procedure.*

4.1 Sampling.

4.1.1 Preparation of collection train. Measure 15 ml of 80 percent isopropanol into the midget bubbler and 15 ml of 3 percent hydrogen peroxide into each of the first two midget impingers. Leave the final midget impinger dry. Assemble the train as shown in Figure 6–1. Adjust probe heater to operating temperature. Place crushed ice and water around the impingers. Leak check the sampling train just prior to use at the sampling site by placing a vacuum gauge at the inlet to the first impinger and pulling a vacuum of at least 250 mm Hg (10 in. Hg), plugging or pinching off the outlet of the flowmeter, and then turning off the pump. The vacuum shall remain stable for at least one minute. Carefully release the vacuum gauge before releasing the flowmeter end. Connect the probe.

4.1.2 Sample collection. Record the initial dry gas meter reading and barometric pressure. To begin sampling, position the tip of the probe at the sampling point and start the pump. Adjust the sample flow to a rate of approximately 1 lpm as indicated by the rotameter. Sample at a rate that is proportional (within 20 percent of the average

$$V_m/\sqrt{\Delta P})$$

to the stack gas velocity throughout the run. Take readings (dry gas meter, temperatures at dry gas meter and at impinger outlet, rate meter, and velocity head) at least every five minutes and when significant changes (20 percent variation in velocity head readings) in stack conditions necessitate additional adjustments in sample flow rate. Add more ice during the run to keep the temperature of the gases leaving the last impinger at 20° C (68° F) or less. At the conclusion of each run, turn off the pump, remove probe from the stack, and record the final readings. Conduct a leak check as before. If excessive leakage rate is found void the test run. Remove the probe from the stack and disconnect it from the train. Drain the ice bath and purge the remaining part of the train by drawing clean ambient air through the system for 15 minutes at the sampling rate.

NOTE.—Clean ambient air can be provided by passing air through a charcoal filter or through an extra midget impinger with 15 ml 3 percent H_2O_2. The tester may option to simply use the ambient air.

4.2 Sample recovery. Disconnect the impingers after purging. Discard the contents of the midget bubbler. Pour the contents of the midget impingers into a leak-free polyethylene bottle for shipment. Rinse the three midget impingers and the connecting tubes with deionized, distilled water and add the washings to the same storage container. Mark the fluid level. Seal and identify the sample container.

4.3 Sample analysis. Note level of liquid in container and confirm whether or not any sample was lost during shipment by noting this on analytical data sheet.

NOTE.—Protect the 0.01 N barium perchlorate solution from evaporation at all times.

Transfer the contents of the storage container to a 100 ml volumetric flask and dilute to exactly 100 ml with deionized, distilled water. Pipette a 20 ml aliquo of this solution into a 250 ml Erlenmeyer flask, add 80 ml of isopropanol, two to four drops of thorin indicator and titrate to a pink endpoint using 0.01 N barium perchlorate. Repeat and average the titration volumes. Run a blank with each series of samples. Replicate titrations shall agree within 1 percent.

5. *Calibration.*

5.1 Use methods and equipment as specified in Methods 2 and 5 and APTD–0576 to

calibrate the rotameter, pitot tube, dry gas meter, barometer, and thermometers.

5.2 Standardize the barium perchlorate solution against 25 ml of standard sulfuric acid to which 100 ml of isopropanol has been added.

6. *Calculations.*

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

6.1 Nomenclature.

C_{SO_2} = Concentration of sulfur dioxide, dry basis corrected to standard conditions, mg/dscm (lb/dscf)
N = Normality of barium perchlorate titrant, milliequivalents/ml
P_{bar} = Barometric pressure at the exit orifice of the dry gas meter, mm Hg (in. Hg)
P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg)
T_m = Average dry gas meter absolute temperature, °K (°R)
T_{std} = Standard absolute temperature, 293° K (528° R)
V_a = Volume of sample aliquot titrated, ml
V_m = Dry gas volume as measured by the dry gas meter, dcm (dcf)
$V_{m(std)}$ = Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm (dscf)
V_{soln} = Total volume of solution in which the sulfur dioxide sample is contained, 100 ml
V_t = Volume of barium perchlorate titrant used for the sample, ml (average of replicate titrations)
V_{tb} = Volume of barium perchlorate titrant used for the blank, ml
32.03 = Equivalent weight of sulfur dioxide

6.2 Dry sample gas volume, corrected to standard conditions.

$$V_{m(std)} = V_m \frac{T_{std}}{T_m} \frac{P_{bar}}{P_{std}} = K \frac{V_m P_{bar}}{T_m}$$

Equation 6–1

where:
K = 0.3855 °K/mm Hg for metric units
= 17.65 °R/in. Hg for English units

6.3 Sulfur dioxide concentration.

$$C_{SO_2} = K \frac{(V_t - V_{tb}) N \left(\frac{V_{soln}}{V_a}\right)}{V_{m(std)}}$$

Equation 6–2

where:
K = 32.03 mg/meq. for metric units
$= 7.05 \times 10^{-5} \frac{(lb)(l)}{(g)(ml)}$ for English units

7. *References.*

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7.3 Matty, R. E. and E. K. Diehl, Measuring Flue-Gas SO_2 and SO_3, Power 101:94–97, November 1957.

7.4 Patton, W. F. and J. A. Brink, Jr., New Equipment and Techniques for Sampling Chemical Process Gases, J. Air Pollution Control Association, 13, 162 (1963).

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METHOD 7—DETERMINATION OF NITROGEN OXIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and Applicability.*

1.1 Principle. A grab sample is collected in an evacuated flask containing a dilute sulfuric acid-hydrogen peroxide absorbing solution, and the nitrogen oxides, except nitrous oxide, are measured colormetrically using the phenoldisulfonic acid (PDS) procedure.

1.2 Applicability. This method is applicable to the measurement of nitrogen oxides emitted from stationary sources. The range of the method has been determined to be 2 to 400 milligrams NO_x as No_2 per dry standard cubic meter without having to dilute the sample.

2. *Apparatus.*

2.1 Sampling (See Figure 7–1).

2.1.1 Probe—Borosilicate glass tubing sufficiently heated to prevent water condensation and equipped with a filter (either in-stack or heated out of stack) to remove particulate matter. Heating is unnecessary if the probe remains dry during the purging period.

2.1.2 Collection flask—Two-liter borosilicate, round bottom with short neck and 24/40 standard taper opening, protected against implosion or breakage.

2.1.3 Flask valve—T-bore stopcock connected to a 24/40 standard taper joint.

2.1.4 Temperature gauge—Dial-type thermometer, or equivalent, capable of measuring 1° C (2° F) intervals from −5 to 50° C (25 to 125° F).

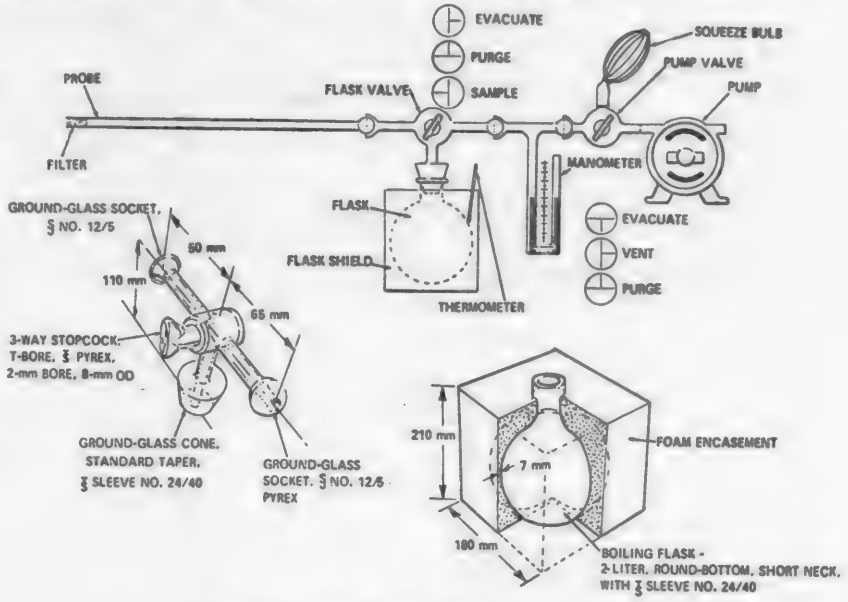

Figure 7-1. Sampling train, flask valve, and flask.

2.1.5 Vacuum line—Tubing capable of withstanding a vacuum of 75 mm Hg (3 in. Hg) absolute pressure, with "T" connection and T-bore stopcock.

2.1.6 Pressure gauge—U-tube monometer, 1-meter, with 1-mm (36-in., with 0.1-in.) divisions, or equivalent.

2.1.7 Pump—Capable of evacuating the collection flask to a pressure equal to or less than 75 mm Hg (3 in. Hg) absolute.

2.1.8 Squeeze bulb—One-way

2.1.9 Volumetric pipette—25-ml.

2.1.10 Stopcock and ground joint grease—A high vacuum, high temperature chlorofluorocarbon grease is required. Halocarbon[1] 25–5S has been found to be effective.

2.1.11 Barometer—Mercury, aneroid, or other barometers capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby weather bureau station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.2 Sample recovery.

2.2.1 Graduated cylinder—50-ml with 1-ml divisions.

2.2.2 Storage container—Leak-free polyethylene bottles.

2.2.3 Wash bottle—polyethylene or glass.

2.2.4 Glass stirring rod.

2.2.5 pH indicating test paper—To cover the pH range of 7–14.

2.3 Analysis.

2.3.1 Volumetric pipettes—Two 1-ml, two 2-ml, one 3-ml, one 4-ml and two 10-ml, and one 25-ml for each sample and standard.

2.3.2 Porcelain evaporating dishes. 175 to 250-ml capacity with lip for pouring, one for

[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

each sample and each standard. The Coors[1] #45006 (shallow-form, 195 ml) has been found to be satisfactory.

2.3.3 Steam bath. (A hot plate is not acceptable.)

2.3.4 Dropping pipette or dropper—Three required.

2.3.5 Polyethylene policeman—One for each sample and each standard.

2.3.6 Graduated cylinder—100-ml with 1-ml divisions.

2.3.7 Volumetric flasks—50-ml (one for each sample), 100-ml (one for each sample, each standard and one for the working standard KNO_2 solution), and one 1000-ml.

2.3.8 Spectrophotometer—To measure absorbance at 410 nm.

2.3.9 Graduated pipette—10-ml, with 0.1-ml divisions.

2.3.10 pH indicating test paper—To cover the pH range of 7–14.

2.3.11 Analytical balance—To measure to 0.1 mg.

3. *Reagents.*

Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available; otherwise, use best available grade.

3.1 Sampling.

3.1.1 Absorbing solution—Cautiously add 2.8 ml concentrated H_2SO_4 to 1 liter of deionized, distilled water. Mix well and add 6 ml of 3 percent hydrogen peroxide, freshly prepared from 30 percent hydrogen peroxide solution. The solution should be used within one week of its preparation. Do not expose to extreme heat or direct sunlight.

3.2 Sample recovery.

3.2.1 Sodium hydroxide (1N)—Dissolve 40 g NaOH in deionized, distilled water and dilute to 1 liter.

3.2.2 Water—Deionized, distilled to conform to ASTM specifications D1193–72, Type 3.

3.3 Analysis.

3.3.1 Fuming sulfuric acid—15 to 18 percent by weight free sulfur trioxide. Handle with caution.

3.3.2 Phenol—White solid.

3.3.3 Sulfuric acid—Concentrated, 95 percent minimum assay. Handle with caution.

3.3.4 Potassium nitrate—Dried at 105–110° C for a minimum of two hours just prior to preparation of standard solution.

3.3.5 Standard solution—Dissolve exactly 2.1980 g of dried potassium nitrate (KNO_3) in deionized, distilled water and dilute to 1 liter with deionized, distilled water in a 1000-ml volumetric flask. For the working standard solution, dilute 10 ml of the standard solution to 100 ml with deionized distilled water. One ml of the working standard solution is equivalent to 100 μg nitrogen dioxide (NO_2).

3.3.6 Water—Deionized, distilled as in section 3.2.2.

3.3.7 Phenoldisulfonic acid solution—Dissolve 25 g of pure white phenol in 150 ml concentrated sulfuric acid on a steam bath. Cool, add 75 ml fuming sulfuric acid, and heat at 100° C (212° F) for 2 hours. Store in a dark, stoppered bottle.

4. *Procedure.*

4.1 Sampling.

4.1.1 Pipette 25 ml of absorbing solution into a sample flask, retaining a sufficient quantity for use in preparing the calibration standards. Insert the flask valve stopper into the flask with the valve in the "purge" position. Assemble the sampling train as shown in figure 7–1 and place the probe at the sampling point. Make sure that all fittings are tight and leak free, and that all ground glass joints have been properly greased with a high vacuum, high temperature chlorofluorocarbon-based stopcock grease. Turn the flask valve and the pump valve to their "evacuate" positions. Evacuate the flask to 75 mm Hg (3 in. Hg) absolute pressure, or less. Evacuation to a lower pressure (approaching the vapor pressure of water at the existing temperature) is even more desirable. Turn the pump valve to its "vent" position and turn off the pump. Check for leakage by observing the manometer for any pressure fluctuation. (Any variation greater than 10 mm Hg (0.4 in. Hg) over a period of 1 minute is not acceptable, and the flask is not to be used until the leakage problem is corrected. Pressure in the flask is not to exceed 75 mm Hg (3 in. Hg) absolute at the time sampling is commenced.) Record the volume of the flask and valve (V_f), the flask temperature (T_1), and the barometric pressure. Turn the flask valve counterclockwise to its "purge" position and do the same with the pump valve. Purge the probe and the vacuum tube using the squeeze bulb. If condensation occurs in the probe and the flask valve area, heat the probe and purge until the condensation disappears. Then turn the pump valve to its "vent" position. Turn the flask valve clockwise to its "evacuate" position and record the difference in the mercury levels in the manometer. The absolute internal pressure in the flask (P_1) is equal to the barometric pressure less the manometer reading. Immediately turn the flash valve to the "sample" position and permit the gas to enter the flask until pressures in the flask and sample line (i.e., duct, stack) are virtually equal. This will usually require about 15 seconds. A longer period indicates a "plug" in the probe which must be corrected before sampling is continued. After collecting the sample, turn the flask valve to its "purge" position and disconnect the flask from the sampling train. Shake the flask for at least 5 minutes.

4.1.2 If the gas being sampled contains insufficient oxygen for the conversion to NO to NO_2, e.g. an applicable subpart of the standard may require taking a sample of a calibration gas mixture of NO in N_2, then oxygen shall be introduced into the flask to permit this conversion. Oxygen may be introduced into the flask by one of three methods: (1) Before evacuating the sampling flask flush with pure cylinder oxygen (then evacuate flash to 75 mm Hg (3 in. Hg) absolute pressure or less); or (2) inject oxygen into the flask after sampling; or (3) sampling may be terminated with a minimum of 50 mm Hg (2 in. Hg) vacuum remaining in the flask, recording this final pressure and then venting the flask to the atmosphere until the flask pressure is almost equal to atmospheric pressure.

4.2 Sample recovery.

4.2.1 Let the flask set for a minimum of 16 hours and then shake the contents for 2 minutes. Connect the flask to a mercury filled U-tube manometer, open the valve from the flask to the manometer, and record the flash temperature (T_f), the barometric pressure and the difference between the mercury levels in the manometer. The absolute internal pressure in the flask (P_f) is the barometric pressure less the manometer reading. Transfer the contents of the flask to a leak-free polyethylene bottle. Rinse the flask twice with 5-ml portions of deionized, distilled water and add the rinse water to the bottle. Adjust the pH to 9–12 by adding sodium hydroxide (1 N) dropwise (about 25 to 35 drops). Check the pH by dipping a stirring rod into the solution and then touching it to the pH test paper. Remove as little material as possible during this step. Mark the height of the liquid level to determine whether or not leakage occurred during transport. Label container to clearly identify its contents. Seal the container for shipping.

4.3 Analysis.

4.3.1 Note level of liquid in container and confirm whether or not any sample was lost during shipment by noting this on analytical data sheet. Transfer the contents of the shipping container to a 50-ml volumetric flask, rinse the container twice with 5-ml portions of deionized, distilled water, add the rinse water to the flask and dilute to the mark with deionized, distilled water. Mix thoroughly and pipette a 25-ml aliquot into the porcelain evaporating dish. Evaporate the solution to dryness on a steam bath and allow to cool. (Use only a steam bath—a hot plate is not acceptable.) Add 2 ml phenoldisulfonic acid solution to the dried residue and triturate thoroughly with a polyethylene policeman. Make sure the solution contacts all the residue. Add 1 ml deionized, distilled water and four drops of concentrated sulfuric acid. Heat the solution on a steam bath for 3 minutes with occasional stirring. Cool, add 20 ml deionized, distilled water, mix well by stirring and add concentrated ammonium hydroxide dropwise with constant stirring until pH is 10 (as determined by pH paper). If the sample contains solids, filter through Whatman No. 41 filter paper into a 100-ml volumetric flask; rinse the evaporating dish with three 5-ml portions of deionized, distilled water and add these to the filter. Wash the filter with at least three 15-ml portions of deionized, distilled water. Add the filter washings to the contents of the volumetric flask and dilute to the mark with deionized, distilled water. If solids are absent, transfer the solution directly to the 100-ml volumetric flask and dilute to the mark with deionized, distilled water. Mix thoroughly and measure the absorbance at 410 nm using the blank solution as a zero reference. Dilute the sample and the blank with a suitable amount of deionized, distilled water if absorbance exceeds A_4, the absorbance of the 400 μg NO_2 standard (See section 5.3).

5. *Calibration.*

5.1 Flask volume. Assemble the flask and flask valve and fill with water to the stopcock. Measure the volume of water to±10 ml. Number and record the volume on the flask.

5.2 Spectrophotometer calibration. Add 0.0 ml, 1.0 ml, 2.0 ml, 3.0 ml and 4.0 ml of the KNO_2 Working standard solution (1 ml=100 μg NO_2) to a series of five porcelain evaporating dishes. To each, add 25 ml of absorbing solution, 10 ml deionized, distilled water and sodium hydroxide (1 N) dropwise until the pH is 9–12 (about 25 to 35 drops each). Beginning with the evaporation step, follow the analysis procedure of Section 4.3 to collect the data necessary to calculate the calibration factor (Section 5.3). This calibration procedure must be repeated on each day that samples are analyzed.

5.3 Determination of spectrophotometer calibration factor K_c.

[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

$$K_c = 100\,\frac{A_1+2A_2+3A_3+4A_4}{A_1^2+A_2^2+A_3^2+A_4^2}$$

Equation 7–1

where:

K_c = Calibration factor
A_1 = Absorbance of the 100 μg NO_2 standard
A_2 = Absorbance of the 200 μg NO_2 standard
A_3 = Absorbance of the 300 μg NO_2 standard
A_4 = Absorbance of the 400 μg NO_2 standard

5.4 Barometer. Calibrate against a mercury barometer.

5.5 Temperature gauge. Calibrate dial thermometers against mercury-in-glass thermometers.

6. *Calculations.*

Carry out the calculations, retaining at lease one extra decimal figure beyond that of the acquired data. Round off figures after final calculations.

6.1 Nomenclature.

A = Absorbance of sample
C = Concentration of NO_x as NO_2, dry basis, corrected to standard conditions, mg/dscm (lb/dscf)
F = Dilution factor (i.e., 25/5, 25/10, etc, required only if sample dilution was needed to reduce the absorbance into the range of calibration)
K_c = Spectrophotometer calibration factor
m = Mass of NO_x as NO_2 in gas sample, μg
P_f = Final absolute pressure of flask, mm Hg (in. Hg)
P_i = Initial absolute pressure of flask, mm Hg (in. Hg)
P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg)
T_f = Final absolute temperature of flask, °K (°R)
T_i = Initial absolute temperature of flask, °K (°R)
T_{std} = Standard absolute temperature, 293° K (528° R)
V_{sc} = Sample volume at standard conditions (dry basis), ml
V_f = Volume of flask and valve, ml
V_a = Volume of absorbing solution, 25 ml
2 = 50/25, the aliquot factor. (If other than a 25-ml aliquot was used for analysis, the corresponding factor must be substituted.)

6.2 Sample volume, dry basis, corrected to standard conditions.

$$V_{sc} = \frac{T_{std}}{P_{std}}\,(V_f - V_a)\left[\frac{P_f}{T_f} - \frac{P_i}{T_i}\right]$$

$$= K(V_f - 25\text{ ml})\left[\frac{P_f}{T_f} - \frac{P_i}{T_i}\right]$$

Equation 7–2

Where:

$$K = 0.3855\,\frac{°K}{\text{mm Hg}} \text{ for metric units}$$

$$= 17.65\,\frac{°R}{\text{in. Hg}} \text{ for English units}$$

6.3 Total μg NO_2 per sample.

$$m = 2K_cAF \quad \text{Equation 7–3}$$

NOTE.—If other than a 25-ml aliquot is used for analyses, the factor 2 must be substituted by a corresponding factor.

6.4 Sample concentration, dry basis, corrected to standard conditions.

$$C = K\,\frac{m}{V_{sc}} \quad \text{Equation 7–4}$$

where:

$$K = 10^3\,\frac{(\text{ml})\ (\text{mg})}{(\text{m}^3)\ (\mu\text{g})} \text{ for metric units}$$

$$= 6.243\times10^{-5}\,\frac{\text{lb/scf}}{\mu\text{g/ml}} \text{ for English units}$$

7. *References.*

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METHOD 8—DETERMINATION OF SULFURIC ACID MIST AND SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and Applicability.*

1.1 Principle. A gas sample is extracted isokinetically from the stack. The acid mist (including sulfur trioxide) and the sulfur dioxide are separated and both fractions are measured separately by the barium-thorin titration method.

1.2 Applicability. This method is applicable for the determination of sulfuric acid mist (including sulfur trioxide) in the absence of other particulate matter and for sulfur dioxide from stationary sources. Collaborative tests have shown that the minimum detectable limits of the method are 0.05 mg/m³ (0.03×10^{-7} lb/ft³) for sulfur trioxide and 1.2 mg/m³ (0.74×10^{-7} lb/ft³) for sulfur dioxide. No upper limits have been established.

2. *Apparatus*

2.1 Sampling. A schematic of the sampling train used in this method is shown in Figure 8–1; it is similar to the Method 5 train except that the filter position is different and heating of the filter holder is not required. Commercial models of this train are available. However, if one desires to build his own, complete construction details are described in APTD–0581; for changes from the APTD–0581 document and for allowable modifications to Figure 8–1, see the following subsections.

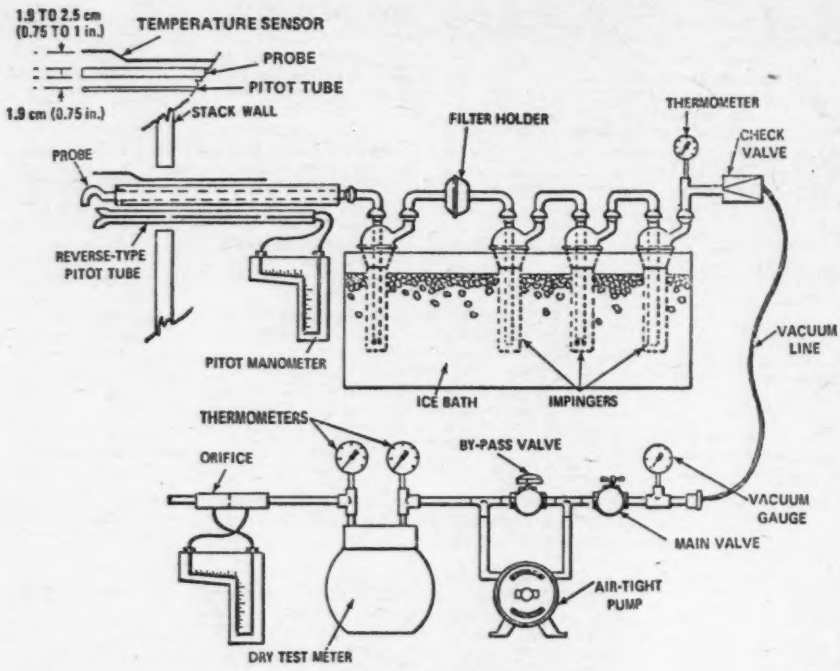

Figure 8 1. Sulfuric acid mist sampling train.

The operating and maintenance procedures for the sampling train are described in APTD–0576. Since correct usage is important in obtaining valid results, all users should read the APTD–0576 document and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein. Further details and guidelines on operation and maintenance are given in Method 5 and should be read and followed whenever they are applicable.

2.1.1 Probe nozzle—Stainless steel (316) with sharp, tapered leading edge. The angle of taper shall be ≤ 30° and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. The nozzle shall be constructed from seamless stainless steel tubing. Other configurations and construction material may be used with approval from the Administrator.

A range of sizes suitable for isokinetic sampling should be available, e.g., 0.32 cm (⅛ in.) up to 1.27 cm (½ in.) (or larger if higher volume sampling trains are used) inside diameter (ID) nozzles in increments of 0.16 cm (1/16 in.). Each nozzle shall be calibrated according to the procedures outlined in the calibration section.

2.1.2. Probe liner—Borosilicate or quartz glass, with a heating system to prevent visible condensation during sampling.

2.1.3 Pitot tube—Type S, or other device approved by the Administrator, attached to probe to allow constant monitoring of the stack gas velocity. The face openings of the pitot tube and the probe nozzle shall be adjacent and parallel to each other, not necessarily on the same plane, during sampling. The free space between the nozzle and pitot tube shall be at least 1.9 cm (0.75 in.). The free space shall be set based on a 1.3 cm (0.5 in.) ID nozzle. If the sampling train is designed for sampling at higher flow rates than that described in APTD–0581, thus necessitating the use of larger sized nozzles, the largest sized nozzle shall be used to set the free space.

The pitot tube must also meet the criteria specified in Method 2 and be calibrated according to the procedure in the calibration section of that method.

2.1.4 Differential pressure gauge—Inclined manometer capable of measuring velocity head to within 10 percent of the minimum measured value or ±0.013 mm (0.0005 in.), whichever is greater. Below a differential pressure of 1.3 mm (0.05 in.) water gauge, micromanometers with sensitivities of 0.013 mm (0.0005 in.) should be used. However, micromanometers are not easily adaptable to field conditions and are not easy to use with pulsating flow. Thus, methods or other devices acceptable to the Administrator may be used when conditions warrant.

2.1.5 Filter holder—Borosilicate glass with a glass frit filter support and a silicone rubber gasket. Other materials of construction may be used with approval from the Administrator. The holder design shall provide a positive seal against leakage from the outside or around the filter.

2.1.6 Impingers—Four as shown in Figure 8–1. The first and third shall be of the Greenburg-Smith design with standard tips. The second and fourth shall be of the Greenburg-Smith design, modified by replacing the insert with an approximately 13 mm (0.5 in.) ID glass tube, having an unconstricted tip located 13 mm (0.5 in.) from the bottom of the flask. Similar collection systems, which have been approved by the Administrator may be used.

2.1.7 Metering system—Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3° C (5.4° F), dry gas meter with 2 percent accuracy, and related equipment, or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

2.1.8 Barometer—Mercury, aneroid, or other barometers capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby weather bureau station, in which the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.9 Temperature gauge—Thermometer, or equivalent, to measure temperature of gas leaving impinger train to within 3° C (5° F).

2.2 Sample recovery.

2.2.1 Wash bottles—Polyethylene or glass, 500 ml. (two).

2.2.2 Graduated cylinders—250 ml, 1 liter. (Volumetric flasks may also be used.)

2.2.3 Storage bottles—Leak-free polyethylene bottles, 1000 ml size. (Two for each sampling run.)

2.3 Analysis.

2.3.1 Pipette—Volumetric 25 ml, 100 ml.

2.3.2 Burette—50 ml.

2.3.3 Erlenmeyer flask—250 ml. (One for each sample blank and standard.)

2.3.4 Graduated cylinder—100 ml.

2.3.5 Trip balance—300 g capacity, to measure to ±0.5 g.

2.3.6 Dropping bottle—to add indicator solution, 125 ml size.

3. *Reagents.*

Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available; otherwise use best available grade.

3.1 Sampling.

3.1.1 Filters—Glass fiber filters, without organic binder exhibiting at least 99.95 percent efficiency (≤0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordane with ASTM standard method D 2986–71. Test data from the supplier's quality control program is sufficient for this purpose.

3.1.2. Silica gel—Indicating type, 6–16 mesh. If previously used, dry at 175° C (350° F) for 2 hours. New silica gel may be used as received.

3.1.3 Water—Deionized, distilled, to conform to ASTM specifications D1193–72, Type 3.

3.1.4 Isopropanol, 80 percent—Mix 800 ml of isopropanol with 200 ml of deionized distilled water.

NOTE.—Experience has shown that only A.C.S. grade isopropanol is satisfactory.

3.1.5 Hydrogen peroxide, 3 percent—Dilute 100 ml of 30 percent hydrogen peroxide to 1 liter with deionized, distilled witer. Prepare fresh daily.

3.1.6 Crushed iced.

3.2 Sample recovery.

3.2.1 Water—Deionized, distilled, to conform to ASTM specifications D1193–72, Type 3.

3.2.2 Isopropanol, 80 percent—Mix 800 ml of isopropanol with 200 ml of deionized distilled water.

NOTE.—Experience has shown that only A.C.S. grade isopropanol is satisfactory.

3.3 Analysis.

3.3.1 Water—Deionized, distilled, to conform to ASTM specifications D1193–72, Type 3.

3.3.2 Isopropanol, 100 percent.

3.3.3 Thorin indicator—1-(o-arsonophenylazo)-2-naphthol-3, 6-disulfonic acid, disodium salt, or equivalent. Dissolve 0.20 g in 100 ml of deionized distilled water.

3.3.4 Barium perchlorate (0.01 N)—Dissolve 1.95 g of barium perchlorate trihydrate ($Ba(ClO_4)_2 \cdot 3H_2O$) in 200 ml deionized distilled water and dilute to 1 liter with isopropanol. Standardize with sulfuric acid as in Section 5.2. This solution must be protected against evaporation at all times. ($BaCl_2$ may also be used.)

3.3.5 Sulfuric acid standard (0.01 N)—Purchase or standardize to ±0.0002 N against 0.01 N NaOH which has previously been standardized against primary standard potassium acid phthalate.

4. *Procedure.*

4.1 Sampling.

4.1.1 Pretest preparation—Follow the procedure outlined in Method 5, Section 4.1.1, except that the filter need not be weighed or identified. If the effluent gas is considered to be dry, i.e., moisture free, the silica gel need not be weighed.

4.1.2 Preliminary determinations—Follow the procedure outlined in Method 5, Section 4.1.2.

4.1.3 Preparation of collection train—Follow the procedure outlined in Method 5, Section 4.1.2.

4.1.3 Preparation of collection train—Follow the procedure outlined in Method 5, Section 4.1.3, except for the second paragraph and use Figure 8–1 instead of Figure 5–1. Replace the second paragraph with: Place 100 ml of 80 percent isopropanol in the first impinger, 100 ml of 3 percent hydrogen peroxide in both the second and third impingers, and about 200 g of silica gel in the fourth impinger. Retain a portion of the reagents for use as blank solutions.

4.1.4 Leak-check procedure—Follow the procedure outlined in Method 5, Section 4.1.4, except that the probe heater shall be adjusted to the minimum temperature required to prevent condensation.

4.1.5 Train operation—Follow the procedure outlined in Method 5, Section 4.1.5, except record the data required on the example sheet shown in Figure 8–2. During the sampling period, observe the line between the probe and the first impinger for signs of condensation. If it occurs, adjust the probe heater setting upward to the minimum temperature required to prevent condensation. After turning off the pump and recording the final readings at the conclusion of each run, remove the probe from the stack and disconnect it from the train. Drain the ice bath and purge the remaining part of the train by drawing clean ambient air through the system for 15 minutes at the average flow rate used for sampling.

NOTE.—Clean ambient air can be provided by passing air through a charcoal filter.

4.2 Sample recovery.

4.2.1 Container No. 1—Transfer the contents of the first impinger to a 250 ml graduated cylinder. Rinse the probe, first impinger, and all connecting glassware before the filter with 80 percent isopropanol. Add the rinse solution to the cylinder. Dilute to 250 ml with 80 percent isopropanol. Add the filter to the solution, mix, and transfer to the storage container. Protect the solution against evaporation. Mark the level of liquid on container and identify the sample container.

4.2.2 Container No. 2—Transfer the solutions from the second and third impingers to a 1000 ml graduated cylinder. Rinse all glassware between the filter and silica gel impinger with deionized, distilled water and add this rinse water to the cylinder. Dilute to a volume of 1000 ml with deionized, distilled water. Transfer the solution to a storage container. Mark the level of liquid on container. Seal and identify the sample container.

PLANT _____
LOCATION _____
OPERATOR _____
DATE _____
RUN NO. _____
SAMPLE BOX NO. _____
METER BOX NO. _____
METER ΔH@ _____
C FACTOR _____
PITOT TUBE COEFFICIENT, Cp _____

SCHEMATIC OF STACK CROSS SECTION

AMBIENT TEMPERATURE _____
BAROMETRIC PRESSURE _____
ASSUMED MOISTURE, % _____
PROBE LENGTH, m (ft) _____
NOZZLE IDENTIFICATION NO. _____
AVERAGE CALIBRATED NOZZLE DIAMETER, cm (in.) _____
PROBE HEATER SETTING _____
LEAK RATE, m^3/min (cfm) _____
PROBE LINER MATERIAL _____

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	STATIC PRESSURE (P_S), mm Hg (in Hg)	STACK TEMPERATURE (T_S) °C (°F)	VELOCITY HEAD (ΔP_S),	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER mm H_2O (in. H_2O)	GAS SAMPLE VOLUME m^3 (ft^3)	GAS SAMPLE TEMPERATURE AT DRY GAS METER		TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °C (°F)
							INLET °C (°F)	OUTLET °C (°F)	
TOTAL							Avg.	Avg.	
AVERAGE							Avg.		

Figure 8-2. Field data.

4.3 Analysis.

Note level of liquid in containers 1 and 2 and confirm whether or not any sample was lost during shipment by noting this on analytical data sheet.

4.3.1 Container No. 1—Shake the container holding the isopropanol solution and the filter. If the filter breaks up, allow the fragments to settle for a few minutes before removing a sample. Pipette a 100 ml aliquot of this solution into a 250 ml Erlenmeyer flask, add 2 to 4 drops of thorin indicator, and titrate to a pink endpoint using 0.01 N barium perchlorate. Repeat the titration with a second aliquot of sample and average the titration values. Replicate titrations should agree within 1 percent.

4.3.2 Container No. 2—Throughly mix the solution in the container holding the contents of the second and third impingers. Pipette a 10 ml aliquot of sample into a 250 ml Erlenmeyer flask. Add 40 ml of isopropanol, 2 to 4 drops of thorin indicator, and titrate to a pink endpoint using 0.01 N barium perchlorate. Repeat the titration with a second aliquot of sample and average the titration values. Replicate titrations should agree within 1 percent.

4.3.3 Blanks—Prepare blanks by adding 2 to 4 drops of throin indicator to 100 ml of 80 percent isopropanol. Titrate the blanks in the same manner as the samples.

5. *Calibration.*

5.1 Use methods and equipment as specified in Methods 2 and 5 and APTD–0576 to calibrate the orifice meter, pitot tube, dry gas meter, thermometers, and barometer.

5.2 Standardize the barium perchlorate solution with 25 ml of standard sulfuric acid, to which 100 ml of isopropanol have been added.

6. *Calculations.*

NOTE.—Carry out calculations retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

6.1 Nomenclature.

A_n = Cross sectional area of nozzle, m^2 (ft^2)
B_{ws} = Water vapor in the gas stream, proportion by volume
$C_{H_2SO_4}$ = Sulfuric acid (including SO_3) concentration, g/dscm (lb/dscf)
C_{SO_2} = Sulfur dioxide concentration, g/dscm (lb/dscf)
I = Percent of isokinetic sampling
N = Normality of barium perchlorate titrant, g. equiv/liter
P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg)
P_s = Absolute stack gas pressure, mm Hg (in. Hg)
P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg)
T_m = Absolute average dry gas meter temperature (see Figure 8–2), °K (°R)
T_s = Absolute average stack gas temperature (see Figure 8–2), °K (°R)
T_{std} = Standard absolute temperature, 293° K (528° R)
V_a = Volume of sample aliquot titrated, 100 ml for H_2SO_4 and 10 ml for SO_2
V_{lc} = Total volume of liquid collected in impingers and silica gel (see Figure 8–2), ml
V_m = Volume of gas sample as measured by dry gas meter, dcm (dcf)
$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter corrected to standard conditions, dscm (dscf)
v_s = Stack gas velocity, calculated by Method 2, Equation 2–7 using data obtained from Method 8, m/sec (ft/sec)
V_{soln} = Total volume of solution in which the sulfuric acid or sulfur dioxide sample is contained, 250 ml or 1000 ml, respectively
V_t = Volume of barium perchlorate titrant used for the sample, ml
V_{tb} = Volume of barium perchlorate titrant used for the blank, ml
θ = Total sampling time, min
13.6 = Specific gravity of mercury
60 = Sec/min
100 = Conversion to percent

6.2 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 8–2).

6.3 Dry gas volume. Correct the sample volume measured by the dry gas meter to standard conditions (20° C and 760 mm Hg or 68° F and 29.92 in. Hg) by using Equation 8–1.

$$V_{m(std)} = V_m \frac{T_{std}}{T_m} \frac{P_{bar} + \frac{\Delta H}{13.6}}{P_{std}} = KV_m \frac{P_{bar} + \Delta H/13.6}{T_m}$$

Equation 8–1

where:

K = 0.3855 °K/mm Hg for metric units

= 17.65 °R/in. Hg for English units

6.4 Volume of water vapor and moisture content. Use Equation 5–2 and 5–3 of Method 5. If the effluent gas is considered to be dry, these calculations need not be carried out.

6.5 Sulfuric acid (including SO_3) concentration.

$$C_{H_2SO_4} = K \frac{N(V_t - V_{tb}) \frac{V_{soln}}{V_a}}{V_{m(std)}}$$

Equation 8–2

where:

K = 0.04904 g/milliequivalent for metric units

$= 1.08 \times 10^{-4} \frac{(lb)\ (l)}{(g)\ (ml)}$ for English units

6.6 Sulfur dioxide concentration:

$$C_{SO_2} = K \frac{N(V_t - V_{tb}) \frac{V_{soln}}{V_a}}{V_{m(std)}}$$

Equation 8–3

where:

K = 0.03203 g/milliequivalent for metric units

$= 7.05 \times 10^{-5} \frac{(lb)\,(l)}{(g)(m)}$ for English units

6.7 Isokinetic variation.

6.7.1 Calculations from raw data.

$$I = \frac{100T_s[KV_{lc} + (V_m/T_m)\ (P_{bar} + \Delta H/13.6)]}{60\theta V_s P_s A_n}$$

Equation 8–4

where:

K = 0.00346 mm Hg-m^3/ml-°K for metric units

= 0.00267 in. Hg-ft^3/ml-°R for English units

6.7.2 Calculations from intermediate values.

$$I = \frac{T_s V_{m(std)} P_{std} 100}{T_{std} v_s \theta A_n P_s 60(1 - B_{ws})}$$

$$= K \frac{T_s V_{m(std)}}{P_s v_s A_n \theta (1 - B_{ws})}$$

Equation 8–5

where:

K = 4.323 for metric units
= 0.0944 for English units

6.8 Acceptable results. If 90 percent ≤ I ≤ 110 percent, the results are acceptable. If the results are low in comparison to the standards and I is beyond the acceptable range, the Administrator may option to accept the results. Use reference 7.4 of Method 5 to make judgments. Otherwise, reject the results and repeat the test.

7. *References.*

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[FR Doc.76–16096 Filed 6–7–76;8:45 am]